



Catalytic hydrotreating of solvent refined coal (SRC-II)
by An-Gong Yeh

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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Abstract:

Solvent Refined Coal (SRC-II) from Pittsburg and Midway Coal Mining Company's pilot plant was hydrotreated with commercial and Montana State University developed catalysts. Twenty-two batch autoclave runs and twenty-five continuous trickle bed reactor runs were performed.

The liquid products were analyzed for sulfur and nitrogen content, and the extent of hydrocracking was determined by ASTM-D86 distillation test.

Nitrogen and sulfur content was decreased to meet the requirements, 0.3wt%. The catalyst lasted three hours before carbon laid down on the preheat section caused shut-down.

The study of catalyst- base properties and metal loading was performed to determine the effects of pore diameter, pore volume, surface area, and MoO₃ concentration on the nitrogen removal. The higher surface area gave the better nitrogen removal. However, the smallest surface area with a large median pore diameter was not the poorest performer. An optimum combination of proper surface area and pore diameter seems important. The effect of MoO₃ concentration on nitrogen removal is dependent on the catalyst base used, but it is insignificant compared with the effect of catalyst base. The larger pore volume base gave the higher liquid product yield.

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An - Gong Yeh

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by

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A thesis submitted in partial fulfillment
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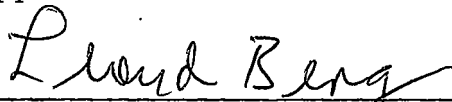
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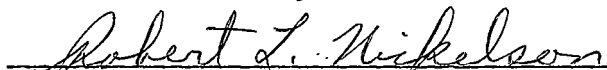
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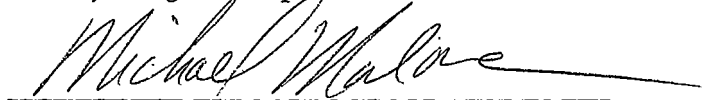
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ABSTRACT

Solvent Refined Coal (SRC-II) from Pittsburgh and Midway Coal Mining Company's pilot plant was hydrotreated with commercial and Montana State University developed catalysts. Twenty-two batch autoclave runs and twenty-five continuous trickle bed reactor runs were performed.

The liquid products were analyzed for sulfur and nitrogen content, and the extent of hydrocracking was determined by ASTM-D86 distillation test.

Nitrogen and sulfur content was decreased to meet the requirements, 0.3wt%. The catalyst lasted three hours before carbon laid down on the preheat section caused shut-down.

The study of catalyst base properties and metal loading was performed to determine the effects of pore diameter, pore volume, surface area, and MoO_3 concentration on the nitrogen removal. The higher surface area gave the better nitrogen removal. However, the smallest surface area with a large median pore diameter was not the poorest performer. An optimum combination of proper surface area and pore diameter seems important. The effect of MoO_3 concentration on nitrogen removal is dependent on the catalyst base used, but it is insignificant compared with the effect of catalyst base. The larger pore volume base gave the higher liquid product yield.

INTRODUCTION

In view of energy crisis and national energy policy, it seems clear that sooner or later the United States will come to rely much more on coal as a resource of energy than it has over the past few decades. It is estimated that coal accounts for 80 percent of the fossil-fuel resources in the U.S.(1). In contrast, for the past decade or so the sources of energy in the U.S. have been predominantly oil and gas (44 and 31 percent respectively), with coal accounting for 21 percent(2). Coal is not the ideal fuel both because it is not a fluid and causes air pollution. Therefore, the development of a technology that will convert the U.S.'s abundant reserves of coal to clean fluid fuels is needed.

Coal conversion processes include gasifications and liquefactions. Since the shortage of domestic liquid hydrocarbons has caused the balance-of-payments problem in the U.S., coal liquefaction schemes are being examined closely. There are three major ways to turn coal into liquid fuels : pyrolysis, indirect liquefaction and direct hydroliquefaction. So far, most pyrolysis

processes haven't been too suitable for making liquid fuels. Although indirect coal technology is in a more advanced state of development, direct hydroliquefaction offers, at least in theory, better economics and higher efficiency in terms of liquids per ton of coal. Therefore, most federal support is going to the direct processes.

Several direct hydroliquefaction processes have been developed such as the Solvent Refined Coal (SRC) process, the Exxon Donor Solvent (EDS) process and the H-Coal process. SRC process is the oldest of these modern processes dating back to 1962. Its original process is known as SRC-I, a later modified version is SRC-II process. Conceivably, its commercial scale plant could be in operation by 1989 or 1990(1).

The product of SRC-II process still cannot be used as a clean fuel at present costs, it must be catalytically upgraded or hydrotreated(3). This research is the second step of SRC-II process. The SRC-II product must be catalytically hydrotreated in a trickle bed reactor to remove unfavorable hetroatom molecules, sulfur and

especially nitrogen, and to improve the overall product. This research is expected to provide the technology for the rapid commercialization of the SRC-II process and give the SRC-II process greater advantages over other processing schemes.

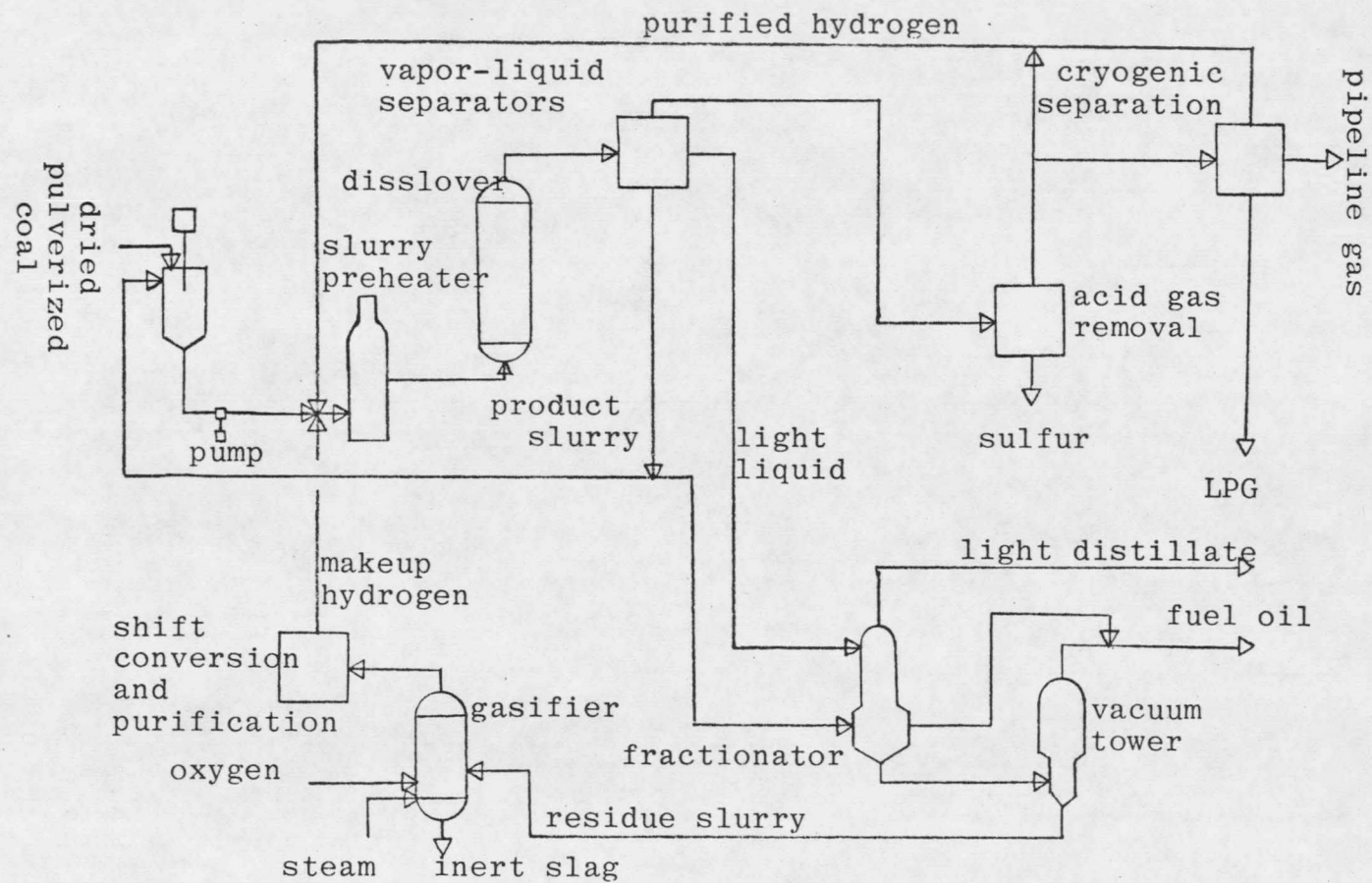
BACKGROUND

SRC-II Process

Of major concern to this research is the SRC-II process operated by Pittsburgh and Midway Coal Mining Company. A fifty ton per day pilot plant is being operated at Fort Lewis, Washington. Pulverized raw coal is mixed with a process-derived slurry product and hydrogen at high temperature and pressure. The coal dissolves; most of its ash and much of its sulfur settle out and can be removed by filtration. Most of the coal is converted to liquids; naphtha, boiler fuel and vacuum residue. This residue contains heavy oil, ash, and undissolved organic material from coal(4). A schematic diagram of the SRC-II process is shown in Figure 1(5).

Chemical Properties of SRC-II Products

The SRC process is not defined as a single product process. The gas and liquid yields per ton of Solvent Refined Coal is shown in Table I(6). Table II(7) presents the analysis of SRC-II product obtained in this research. The SRC-II product shown was made from



5

FIGURE 1. SRC-II PROCESS SCHEMATIC

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 C₁ - C₄ gas cut:

	<u>Vol. %</u>	<u>BTU value/ft³</u>
CH ₄	67.0	680
C ₂ H ₆	19.3	340
C ₃ H ₈	10.0	260
C ₄ H ₁₀	3.7	120
	<u>100.0</u>	<u>1400</u>

* Per ton solvent refined coal

TABLE II
SRC-II Vacuum Flash Feed as Received

=====

Sampled 1-24-77

% Carbon	87.43
% Hydrogen	7.15
% Nitrogen	1.17
% Sulfur	0.72
% Oxygen	3.72
% Ash	0.249

ASTM D-86 Distillation @ 640 mmHg

<u>Volume, ml</u>	<u>Temperature, °F</u>
IBP	408
5	445
10	485
15	544
20	598
25	642
30	684
33.5	Final

wt% recovered 69.2; Volume% recovered 69.7

=====

Kentucky #9 from the Colonial Mine. The analysis of Kentucky #9 coal is shown in Table III.

The Chemistry of Catalytic Hydrotreating

Catalytic hydrotreating of petroleum and coal liquids consists of two main parts: the hydrogenation of unsaturated hydrocarbons and the hydrogenolysis of heteromolecules. Usually hydrocracking also occurs at some of the more severe process conditions. The hydrogenation, desulfurization and denitrogenation play important roles in this research.

If SRC-II product is to be used as a boiler fuel; a hydrogen to carbon atoms ratio of about 2:1, and nitrogen, sulfur and mineral level below 0.5 wt% is required. The sulfur level is determined from the current Environmental Protection Agency (EPA) Standards(8). If the SRC-II product is to be a feed stock for a conventional catalytic cracker, the nitrogen requirement is much more stringent. Catalysts in catalytic cracking operations provide acid sites which facilitate cracking of hydrocarbon feeds. If nitrogen is present, it neutralizes these acid sites

TABLE III

SRC Feed Coal Analysis, January 1977

=====

Kentucky #9 Coal

	<u>wt%</u>
Carbon	71.35
Hydrogen	5.07
Nitrogen	1.44
Sulfur	3.50
O ₂	7.55
Ash	10.12
Moisture	0.97

Sulfur Forms (wt% on Coal)

Pyritic sulfur	1.63%
Sulfate Sulfur	0.09%
Organic Sulfur	<u>1.76%</u>
Total	3.48%

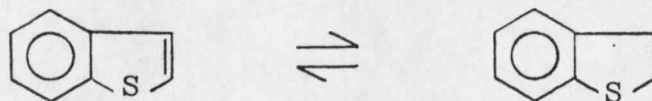
Average Mineral Residue Analysis (wt%)

Carbon	27.61%
Hydrogen	1.39%
Nitrogen	0.54%
Sulfur	7.29%
Ash	63.17%
Pyridine Insol	96.98%

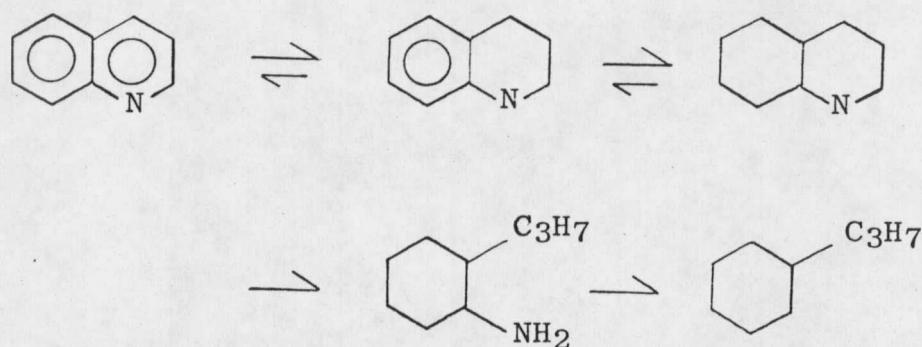
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and acts as a poison. Deactivation of the catalyst progresses in direct proportion to the duration of operation and in proportion to the amount of nitrogen in the catalytic cracker feed. The preferred nitrogen level of catalytic cracker feed is in the range of 100-400 ppm(9). Several hydrocracking processes can tolerate nitrogen levels of 0.3 wt% in the feedstock. Examples of these processes are Standard Oil's Ultracracking process and Union Oil's Unicracking process(10,11).

Several sulfur and nitrogen compounds such as benzothiophenes and quinolines, which make it more difficult for the desulfurization and denitrogenation of coal liquids, have been studied in microreactors to give an insight into the mechanisms(12-16). In the hydrodesulfurization of benzothiophene, it was found that the hydrogenation of the double bond in the thiophene ring took precedence over the removal of sulfur.



Benzothiophene and benzohydrothiophene 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). Methyl substitution near the sulfur atom in dibenzothiophenes greatly reduces the hydrogenation activity, believed to be caused by steric effects which restricts the interaction of the sulfur atom and the surface of the catalyst(12,14,15). Usually nitrogen is more difficult to remove than sulfur from hydrocarbon streams. Conditions which reduce excess nitrogen content to a satisfactory level will usually effectively remove excess sulfur. It has been shown that the total rate of hydrodenitrogenation shows a maximum with respect to hydrogen partial pressure. However, 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(14,15,16).



Hydrotreating Catalysts

Since petroleum hydrotreating processes are governed by fairly well established kinetics, the most important factor for further progress in the coal liquefaction is the catalyst. Traditionally, a hydrotreating catalyst consists of an active component, usually a metal, that is deposited on a high surface area support. The support is considered inert and its purpose is not only to disperse the metal component, but also to provide acid sites to initiate the carbonium ion mechanisms of cracking reaction(17). Generally, pure silica is less acid than

alumina, which is less acid than silica-alumina. The metals on the support also influence acidity. It was found that NiO on alumina reduces the acidity, while MoO₃ increases it(18).

In catalytic hydrotreating, one of reasons for catalyst deactivation is the deposition of carbonaceous materials(19). It is believed that the Solvent Refined Coal has a high asphaltene content with an average size of 40-50 Å per molecule and smaller pores of the catalyst tend to plug up. The large molecule causes the problem of pore diffusion limitation. Therefore, the effects of surface area and pore size must be accounted for. Theoretically, the higher surface area gives the higher initial activity of catalyst and the larger pore diameter obtains a longer catalyst life(20). In an attempt to develop a satisfactory catalyst, high surface area and/or large pore diameter bases were used in this research.

The most common metals responsible for the hydrogenation-dehydrogenation function of a hydrotreating

catalyst are molybdenum and tungsten. The metals Ni, Co, Fe, Zn, and Cr are usually described as promoters. The function of the promoter is believed to increase the number of exposed molybdenum or tungsten ions-the active centers for the hetro-atom removal reaction(21). For cobolt-moly catalysts, the ratio is about 1:3. This has been found to be independent of support and material(22). The fabrication of catalysts with Mo, W, Ni, and Co or these combinations is the basis of this research.

Operation Conditions of Trickle Bed Reactor

In the simplest terms the conversion of coal into oil or gas calls for adding hydrogen. The ratio of hydrogen atoms to the carbon atoms in coal is about 0.87:1. The consumption of hydrogen is a major cost in the conversion of coal into oil. An optimal hydrogen flow rate of 10,000 scf/bbl investigated by Runnion(23, 24) was used in all catalyst tests in this research.

In trickle bed reactors the catalyst is fixed, the flow pattern is close to plug flow, and liquid to

catalyst ratio is much lower thus limiting side reactions. In the petroleum industry, typically less reactive, higher boiling-viscous feeds are operated at low liquid flow rates. The liquid hourly space velocity (LHSV-Volume of Liquid Feed/(Volume of Catalyst x Hour)) of 1.0 usually was used in this research. Generally speaking, higher space velocities will give lower conversions.

Representative operating conditions for the reactors are a pressure range of 500-2,500 psig and a temperature range of from 345 °C to 425 °C. In most fixed bed reactors, as the run progresses, it is necessary to raise the temperature to compensate for the loss in catalyst activity in order to increase reaction rate and maintain conversion levels. It was found that the higher temperatures give the higher conversions, however, the conversion of hydrocarbon to coke(25) also increases. So there is no good reason to operate at a higher reactor temperature. In the petroleum industry, it is understood that most of the carbon is laid down

in the initial running period, so starting at a lower temperature and then increasing the temperature gradually should prevent the reactor from coking up. However, it was found in coal research(26) that a longer packed bed reactor and higher feed flow rates will give a higher pressure drop caused by carbon lay down on the packed bed. It also has been shown that a spherical support is able to prevent reactor bed plugging(27). A study of effect of pressure on the activity of the catalyst reported that better results can be obtained by using a higher pressure, but 1,000 psig is the limiting working pressure of the equipment in this research(28,29).

RESEARCH OBJECTIVE

This research is an attempt to upgrade the SRC-II product to a feedstock suitable for a conventional refinery or a boiler fuel. The SRC-II product is a tar-like substance received from Pittsburg and Midway Coal Mining Company.

The amount of sulfur and nitrogen are to be reduced and the amount of product yield in ASTMD-86 distillation is to be improved. The reasons for the removal of sulfur and nitrogen is to prevent the catalyst poisoning in further refining steps and to reduce pollution from any eventual fuels made from the SRC-II process. Catalysts, either self-fabricated or commercial manufactured, were to be evaluated in a batch autoclave reactor and continuous trickle bed reactor in this research. The purpose is to determine the best catalyst and the best operation condition for trickle bed reactor.

MATERIALS, EQUIPMENT AND PROCEDURES

Feedstock

The Pittsburgh and Midway Coal Mining Company provided the SRC-II product that was used as feed in this research. SRC-II product was made from Kentucky #9 Coal from the Colonial Mine. The analysis of this coal is listed in Table III. SRC-II product analyses are listed in Table II.

Catalyst Fabrication

All catalysts fabricated at Montana State University were prepared by impregnating commercial supports with metal salts using the incipient wetness technique. The procedure used was as follows :

1. Oven dry the supports at 110 °C for 8 hours
2. Calcine the supports at 450 °C for 8 hours
3. Cool to room temperature in a dessicator
4. Record weight of the support
5. Impregnate the support in a slowly rotating jar with a specific metal solution, the concentration of which is calculated by the formulation(30) :

$$\begin{aligned} & \text{metal oxide percent in the support} \\ &= \text{conc. of solution} \times \text{pore vol.} / \\ & (1 + (\text{pore vol.} \times \text{conc. of solution})) \end{aligned}$$

The concentration of solution is further adjusted by experience.

6. Air dry in an air stream of 3 psig
7. Oven dry the impregnated supports at 110 °C for 8 hours
8. Calcine the impregnated supports at 450 °C for 8 hours
9. Cool to room temperature in a dessicator
10. Record the weight and calculate the weight percent of metal oxide impregnate

This procedure was repeated as needed to obtain the objective percentages of metal oxides.

Catalyst Pretreatment

All catalysts were pretreated by sulfiding. This procedure is used to activate the catalyst and to prevent the reduction of catalyst activity by hot hydrogenation(31,32). The catalyst was treated with a

10% hydrogen sulfide in hydrogen mixture for 12 hours in order to sulfide the metal oxides into the metallic sulfides. The stream of hydrogen sulfide was passed through the pipe reactor at approximately atmospheric pressure. Exit gas from the apparatus was scrubbed with 20% sodium hydroxide-water solution before venting to the hood. Temperature was maintained at 325 °C by use of a powerstat to control an electric pipe heater. Extreme caution should be taken whenever handling hydrogen sulfide because it causes coma and death within a few seconds after a few inspirations. Hydrogen sulfide is extremely hazardous because it fatigues the sense of smell in high concentrations; therefore, it gives no warning(33).

Batch Autoclave Runs

Batch runs were made in Parr Series 4,000 pressure reaction apparatus(34). The apparatus was heated in a rocking autoclave heater. The Parr autoclave and heater-rocker are shown in Figure 2.

The autoclave was charged with 25 ml of catalyst

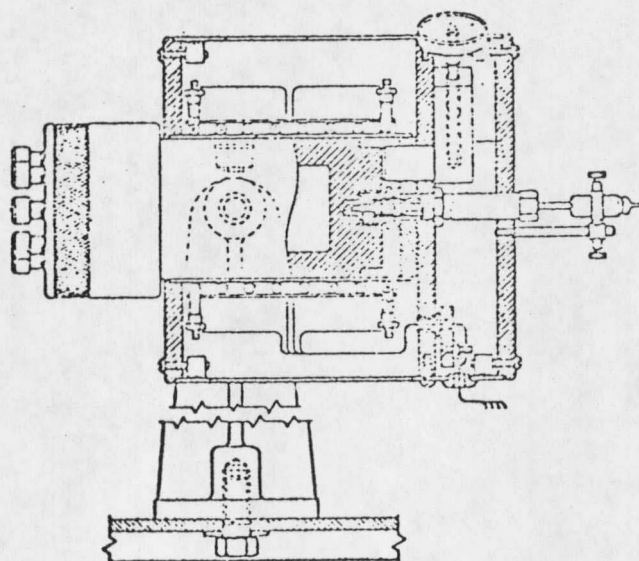
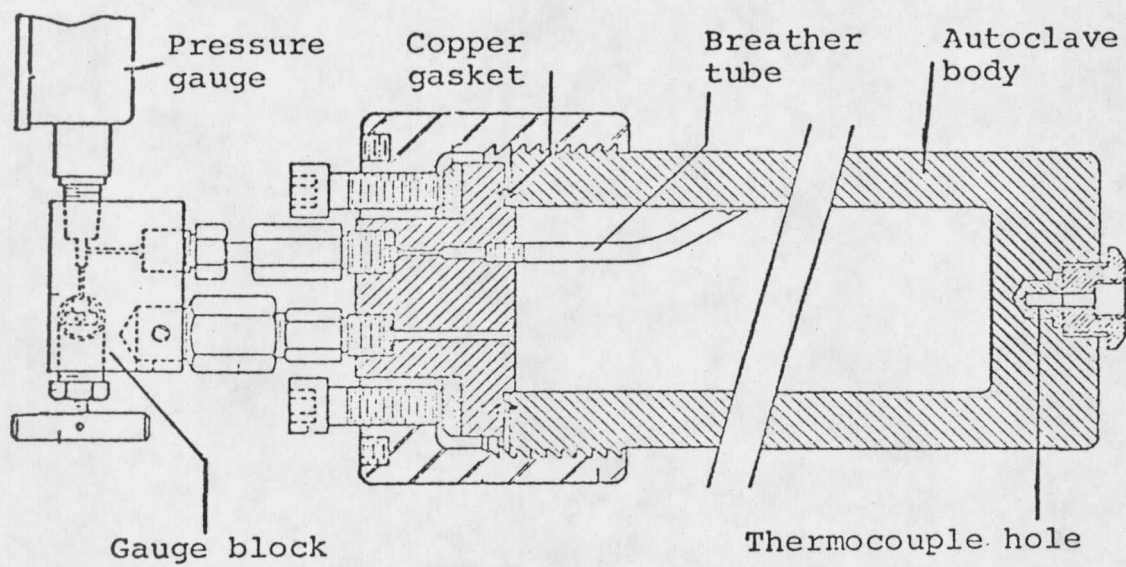


FIGURE 2. ROCKING AUTOCLAVE ASSEMBLY DETAILS

and 200 ml of SRC-II product. The copper head of autoclave and copper gasket were secured using a torque wrench. A new copper gasket was torqued to 60 ft-lb with subsequent 10 ft-lb increases per run. The copper gasket was replaced when a torque of 100 ft-lb was reached. After the head of reactor was secured, the pressure gauge and gauge block was attached to the autoclave head. The autoclave was pressurized with hydrogen to $2,500 \pm 50$ psig using a Haskel gas booster air-driven compressor(35) and checked for leaks. The autoclave was then heated up to 425 ± 5 °C in the heater-rocker(34), which usually took 1.5 hours. The residence time of a run was 1.5 hours. Silver Goop(36) was used on all threaded autoclave connections to prevent bolt seizure at high temperature. An iron-constantan thermocouple, placed in the base of the autoclave, connected to a single point Micromax recorder(37) 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. Then the hydrogen consumption (measured by the difference in cold loading pressure and the final pressure at room temperature) was recorded. The gas in the autoclave was then vented in a hood by opening the needle valve 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.

Continuous Trickle Bed Reactor

The trickle bed reactor was designed and constructed by the Chemical Engineering Department at Montana State University prior to this research. The schematic diagram of the trickle bed reactor is shown in Figure 3.

Two different lengths of reactor were used in this research. One was forty inches long, the other thirty-six inches. Both of them are made by 1-inch

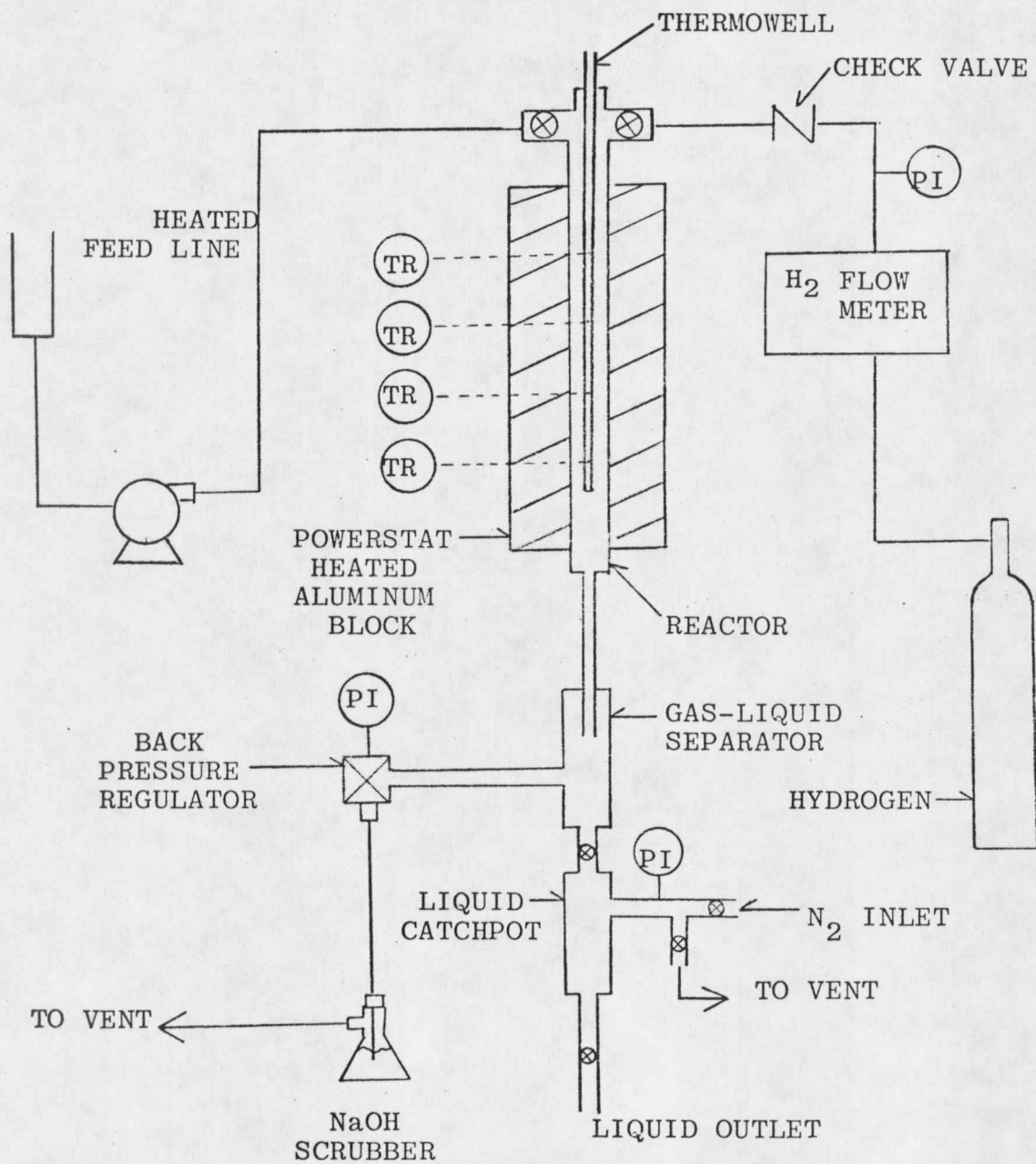


FIGURE 3. TRICKLE BED REACTOR

I.D. schedule-80 Inconel pipe. The top of the reactors are fitted with a 1/4-inch stainless steel cross. This allows the fitting of a 36-inch, or 32-inch, stainless steel tubing, which serves as a thermowell, and the fitting of two feed ports, one for SRC-II feed and one for hydrogen.

The reactors are placed into the 1-inch bore hole of a 6-inch O.D. aluminum block which is about three feet long. The longer reactor extends 6-inch outside the top of aluminum block, the top of shorter reactor and aluminum block are about of equal length. The aluminum block is wrapped with three sets of nichrome wire heating coils encased in ceramic beads. Each heating coil is connected to a Powerstat variable transformer which is manually controlled for temperature. Four iron-constantan thermocouples were placed in the thermowell at six inch intervals. These four thermocouples allow the monitoring of two temperatures in the preheat section of the reactor and two temperatures in the catalyst section of the bed. In the

preheat zone, the longer reactor and shorter reactor were loaded at the top with 175 c.c. and 125 c.c. of 1/4" Denstone support(38) respectively, then followed by 25 c.c. of 1/8" Denstone inert support. The sixty cubic centimeters of catalyst mixed with 60 c.c. of 1/8" inert support was loaded into the catalyst zone. The bottom section of the reactor was filled with 1/8" Denstone inert support. Then a cone of stainless steel screen was inserted as a plug support above the 1/4-inch I.D. reactor closure that was threaded into the pipe. The threaded connection was sealed with Teflon tape and Silver Goop to prevent leakage.

SRC-II product was pumped into the top of the reactor by use of a Milton Roy Model MR-1-49 Simplex packed piston pump through a 1/8" stainless steel feedline. The pump is equipped with a manually controlled micrometer adjustment for feed rate control. All feedlines and reservoirs were wrapped with Cole-Parmer flexible heating cords(39) to prevent the feed from freezing up. Technical grade hydrogen is fed

through a regulator; a micrometer valve, a Brooks Thermal Mass Flowmeter(40), and a ball check valve to the top of the reactor.

Gases and liquids passed through the reactor to a gas-liquid separator. The gases pass through a condenser and through a Grove back pressure regulator. The Grove back pressure regulator was equipped with a Teflon diaphragm to handle the corrosive gases. The exit gases passed through a 20% NaOH-water solution and then was vented. A wet test meter can be connected before the gas is vented in order to calibrate the Brooks Thermal Mass Flowmeter. The liquids passed from the gas-liquid separator into a pressurized catchpot. When a liquid sample was taken, the valve between the separator and the catchpot was closed. The catchpot was then depressurized and the sample was drained from the bottom of the catchpot. The catchpot was then repressurized with nitrogen and the valve was reopened.

Continuous Trickle Bed Runs

After the reactor was loaded as previously des-

cribed, it was placed in the aluminum block. The catch-pot system, and the liquid and hydrogen feedlines were attached. The thermocouples, connected to a Leeds and Northrup Multipoint recorder, were then placed in the thermowell. The whole system except the pump was pressurized and checked for leaks. If no leaks were found, the system was depressurized. The variable Powerstats were then turned on and the system was allowed to heat for ten hours.

When the reactor reached run temperature, the SRC-II product, all liquid feedlines, reservoirs and pump jacket were preheated. The feed reservoir was filled and SRC-II product was pumped through the feed lines. Then the pump was stopped and the feed line connected to the top of the reactor. If the feed line is not filled first, the pump will tend to cavitate on the pressurized system.

The reactor was slowly pressurized with hydrogen. When the system had reached the desired pressure, the by-pass valve on the flowmeter was closed and the micro-

metering valve was adjusted to keep the desired hydrogen flow rate, 10,000 standard cubic feet per barrel (scf/bbl). The valve of the feed line was then opened and the pump started. The liquid flow rates were measured by using a stop watch and timing the flow from a graduated side-arm attached to the main feed reservoir. The flow rate was checked frequently to maintain an even flow and the average flow rates was reported. The flow rates for all runs were kept at a liquid hourly space velocity (LHSV) of one except Run A-2 which was 0.5 LHSV.

For Runs A-1 to A-4, samples were taken every three hours for 12 hours. In Runs A-5 to A-21, only two hour samples or a little longer were collected. Runs A-22 to A-25 were shut down when the reactor pressure reached 1,300 psig. In Runs A-5 to A-25, unless specifically noted, samples were taken as follows :

Time on Stream

minutes :	30	45	60	75	90	105	120	150	180	210	240
sample :	1	2	3	4	5	6	7	8	9	10	11

The amount of samples in Runs A-21 to A-25 were measured by volume. The ratio of sample volume to the volume of feed was reported as the yield of oil. Gaseous products and hold-up in the reactor were not part of the yield.

After the last sample was taken, the pump was shut off, the valve between the feed line and reactor was closed and the feed line was removed. The excess SRC-II product was then drained.

The hydrogen flow and heaters were shut off and the reactor was depressurized. The catchpot system was removed and cleaned thoroughly with acetone. The hydrogen inlet valve at the top of reactor was then closed. The reservoirs were filled with 30 W motor oil, which was pumped through the feed line while the reactor was still hot. The motor oil was to loosen catalyst particles and flush the system of reactants. The smoke from hot motor oil is very hard to tolerate and the room must be vented thoroughly during cleaning. After flushing with motor oil, the reactor was removed from the aluminum block with asbestos gloves. The feed

line and reservoir were cleaned with acetone. After the reactor was cooled to room temperature, catalyst and inert supports were knocked or drilled out. Then the reactor was cleaned with acetone to be used in the next run.

Analytical Procedure

The liquid products from all runs were analyzed for sulfur content, nitrogen content, and the extent of cracking.

Sulfur analysis was done on all samples for continuous Runs A-1 to A-4, and for all batch runs. Sulfur analysis on selected samples was done for continuous Runs A-5 to A-25. The analyses were performed by the quartz tube combustion method using a Bico-Brown Shell design sulfur apparatus(41,42,43). Sulfur content of the feed, SRC-II, is 0.72 wt%. Weight percent desulfurization (% DS) was calculated as follows :

$$(0.72\% - \text{wt\%}) / 0.72\% = \%DS$$

Nitrogen content was determined by Macro-Kjeldahl method(43,44,45) using 0.5 grams of samples and 40 grams of potassium sulfate. SRC-II contains 1.17% nitrogen. Weight percent denitrogenation (%DN) is calculated similarly to %DS.

The extent of cracking was determined by ASTM D-86 atmospheric distillation(46). This technique measures the cumulative amount of product which boils below 700 °F or when decomposition begins, whichever occurs first. The amount of the sample used for the distillation was 50 ml whenever possible.

RESULTS AND DISCUSSION

Twenty-two batch autoclave runs and twenty-five continuous trickle bed runs were performed. Four commercial catalysts and twenty-nine catalysts that were fabricated at Montana State University were tested in these runs. The data for each run are presented in the appendices. Appendix A contains the data from the batch runs which are specified the prefix B. Appendix B contains the data from the continuous runs which are specified the prefix A. All samples taken from all runs were analyzed for nitrogen content and the amount of distillable liquids. Sulfur analyses, for Runs A-5 to A-25, were done only on periodic samples to reduce the load of analytical work. A few samples in Runs A-7 and A-14 were missed due to the inadvertant handling in the process of analysis.

Commercial catalysts used are shown in Table IV. A variety of pore diameter bases in Table V were received from Nalco Chemical Company. Three major effects were to be investigated:

- (1) the effect of the base; pore diameter, pore

TABLE IV

COMMERCIAL CATALYST DESCRIPTION

Catalyst*	Chemical Combination	Surface Area, m ² /g	Pore Volume, ml/g	Ave. Pore Diameter, Å**
HARSHAW HT-400E 1/16"	15%MoO ₃ , 3%CoO	220	.5	91
CYANAMID HDS-20A 1/16", Trilobe	16.2%MoO ₃ , 5%CoO	230	.52	90
NALCO NM 502 1/16"	14%MoO ₃ , 4%NiO	240	.53	88
NALCO MO 477 1/16"	14%MoO ₃ , 3.3%CoO	250	.55	88
HARSHAW HT-400E 1/16" 720A-2-1-1	14.8%MoO ₃ , 2.8%CoO	222	.51	110

* Catalysts are on alumina bases

** Ave. Pore Diameter(Å) = $40,000 \frac{\text{Pore Volume}}{\text{Surface Area}}$

TABLE V
PROPERTIES OF CATALYST BASES

Sample No.	Composition	Surface Area, m ² /g	Pore Vol. ml/g	Median Pore Dia. Å	Average [*] Pore Dia., Å
NALCO-78-6008A-1/32"	4%SiO ₂ , 96%Al ₂ O ₃	323.2	.7183	90.2	88.9
NALCO-78-6008B-1/16"	100%Al ₂ O ₃	232.4	.7215	137.3	124.2
NALCO-78-6008C-1/32"	2%SiO ₂ , 98%Al ₂ O ₃	214.57	.8397	161	156.5
NALCO-78-6008D-1/16"	9%P ₂ O ₅ , 91%Al ₂ O ₃	211.39	.7943	190	150.3
NALCO-78-6008E-1/16"	17%P ₂ O ₅ , 83%Al ₂ O ₃	146.95	.6841	420.2	186.2
NORTON-6176-1/16"	99.85%Al ₂ O ₃ , .12%SiO ₂ , .014%Na ₂ O	250	.70		152

* Average Pore Diameter, Å = 40,000(Pore Vol./Surface Area)

volume and surface area specifically

(2) the effect of MoO_3 content

(3) the effect of additional WO_3

by impregnating the same or nearly the same amount of metal oxides on to each base as well as different metal compositions on to the same base. Table VI presents the actual analyses of the amount of metals loaded and the base carrier material. All catalysts fabricated at Montana State University were designated the prefix MSU. NiO and CoO were kept as constant as possible, the objective content of NiO and CoO, prior to MSU-C24-E, being 0.5% and 0.8% respectively. Catalysts MSU-C25-A to MSU-C29-E were impregnated the same metal composition of 13% MoO_3 3.0%NiO 7.0%CoO 10.0% WO_3 .

Batch Autoclave Runs

Batch tests were performed on SRC-II product to evaluate the activity of catalysts. As continuous runs had been very long, it was thought that the batch runs could provide relatively fast catalyst testing. In an attempt to investigate the activity of catalysts influenced by the base, MoO_3 content and additional WO_3 , twenty-two

TABLE VI
MSU CATALYST DESCRIPTION

Catalyst	%MoO ₃	%NiO	%CoO	%WO ₃	Base
MSU-C1-A	19.4	.1	.43	-	NALCO-78-6008A-1/32"
MSU-C2-A	12.0	.48	.1	-	NALCO-78-6008A-1/32"
MSU-C3-A	10.4	1.16	.11	-	NALCO-78-6008A-1/32"
MSU-C4-A	9.1	.37	1.38	8.2	NALCO-78-6008A-1/32"
MSU-C5-B	18.6	.26	.45	-	NALCO-78-6008B-1/16"
MSU-C6-B	14.8	.19	1.17	-	NALCO-78-6008B-1/16"
MSU-C7-B	14.6	.13	5.8	-	NALCO-78-6008B-1/16"
MSU-C8-B	9.8	.45	.43	-	NALCO-78-6008B-1/16"
MSU-C9-B	8.3	.57	1.55	8.8	NALCO-78-6008B-1/16"
MSU-C10-C	20.6	.1	1.08	-	NALCO-78-6008C-1/32"
MSU-C11-C	11.1	.1	1.56	-	NALCO-78-6008C-1/32"
MSU-C12-C	9.5	.44	1.77	-	NALCO-78-6008C-1/32"
MSU-C13-C	9.05	.79	1.6	9.0	NALCO-78-6008C-1/32"
MSU-C14-D	20.7	1.86	1.2	-	NALCO-78-6008D-1/16"
MSU-C15-D	13.7	.78	.1	-	NALCO-78-6008D-1/16"

TABLE VI(continued)

Catalyst	%MoO ₃	%NiO	%CoO	%WO ₃	Base
MSU-C16-D	11.8	.1	1.9	-	NALCO-78-6008D-1/16"
MSU-C17-D	9.3	.17	1.76	8.8	NALCO-78-6008D-1/16"
MSU-C18-E	29.3	.1	.1	-	NALCO-78-6008E-1/16"
MSU-C19-E	18.9	.1	3.5	-	NALCO-78-6008E-1/16"
MSU-C20-E	13.0	.1	.3	-	NALCO-78-6008E-1/16"
MSU-C21-E	9.5	.37	1.89	-	NALCO-78-6008E-1/16"
MSU-C22-E	9.8	.3	1.4	9.2	NALCO-78-6008E-1/16"
MSU-C23-B	23.0	.2	1.4	-	NALCO-78-6008B-1/16"
MSU-C24-E	15.5	.15	2.3	-	NALCO-78-6008E-1/16"
MSU-C25-A	12.5	2.66	6.65	9.66	NALCO-78-6008A-1/32"
MSU-C26-B	13.2	3.0	6.7	9.45 ,	NALCO-78-6008B-1/16"
MSU-C27-C	11.54	3.16	6.6	9.5	NALCO-78-6008C-1/32"
MSU-C28	12.6	2.87	6.48	7.75	NORTON-6176-1/16"
MSU-C29-E	14.4	3.1	4.5	9.1	NALCO-78-6008E-1/16"

catalysts were tested in the batch autoclave runs, which are designated B-1 to B-22. The detailed data from these tests are presented in Appendix A. Table VII summarizes the catalyst activity on denitrogenation, desulfurization, and distilled yield at 650 °F. Unfortunately, it is hard to determine which base is the best or to obtain a general expression for the effect of composition of catalyst effectiveness from these data. Figures 4, 5, 6, and 7, 8 help to show the tendency of each base for the different amounts of MoO_3 impregnated. Nevertheless some important information still can be obtained from these figures : (1) a better catalytic activity for both desulfurization and denitrogenation can be obtained by increasing the content of MoO_3 on base B (2) increasing the concentration of MoO_3 on base A or decreasing it on bases C and D might yield a better catalyst for desulfurization (3) there is no significant improvement in denitrogenation with additional WO_3 . The distillation results of batch

TABLE VII
BATCH RUN DATA SUMMARY

Run	Catalyst	wt%DS	wt%DN	%Yield*
B-1	MSU-C1-A	55.6	12.0	73
B-2	MSU-C2-A	39.6	21.4	66
B-3	MSU-C3-A	32.6	18.8	60
B-4	MSU-C4-A	47.9	20.1	69
B-5	MSU-C5-B	52.8	19.2	70
B-6	MSU-C6-B	50.0	9.4	64
B-7	MSU-C7-B	52.1	2.6	73
B-8	MSU-C8-B	36.1	19.2	62
B-9	MSU-C9-B	36.8	8.5	65
B-10	MSU-C10-C	20.8	15.4	71
B-11	MSU-C11-C	41.0	12.0	67
B-12	MSU-C12-C	41.7	12.8	66
B-13	MSU-C13-C	62.6	10.3	74
B-14	MSU-C14-D	41.0	15.4	60
B-15	MSU-C15-D	45.1	12.8	74
B-16	MSU-C16-D	56.9	15.4	60
B-17	MSU-C17-D	32.6	8.5	72
B-18	MSU-C18-E	47.9	1.7	64
B-19	MSU-C19-E	20.1	6.0	73
B-20	MSU-C20-E	47.9	22.2	60
B-21	MSU-C21-E	46.0	6.5	70
B-22	MSU-C22-E	51.5	11.1	67

* ASTM volume percent distilled yield at 650 °F

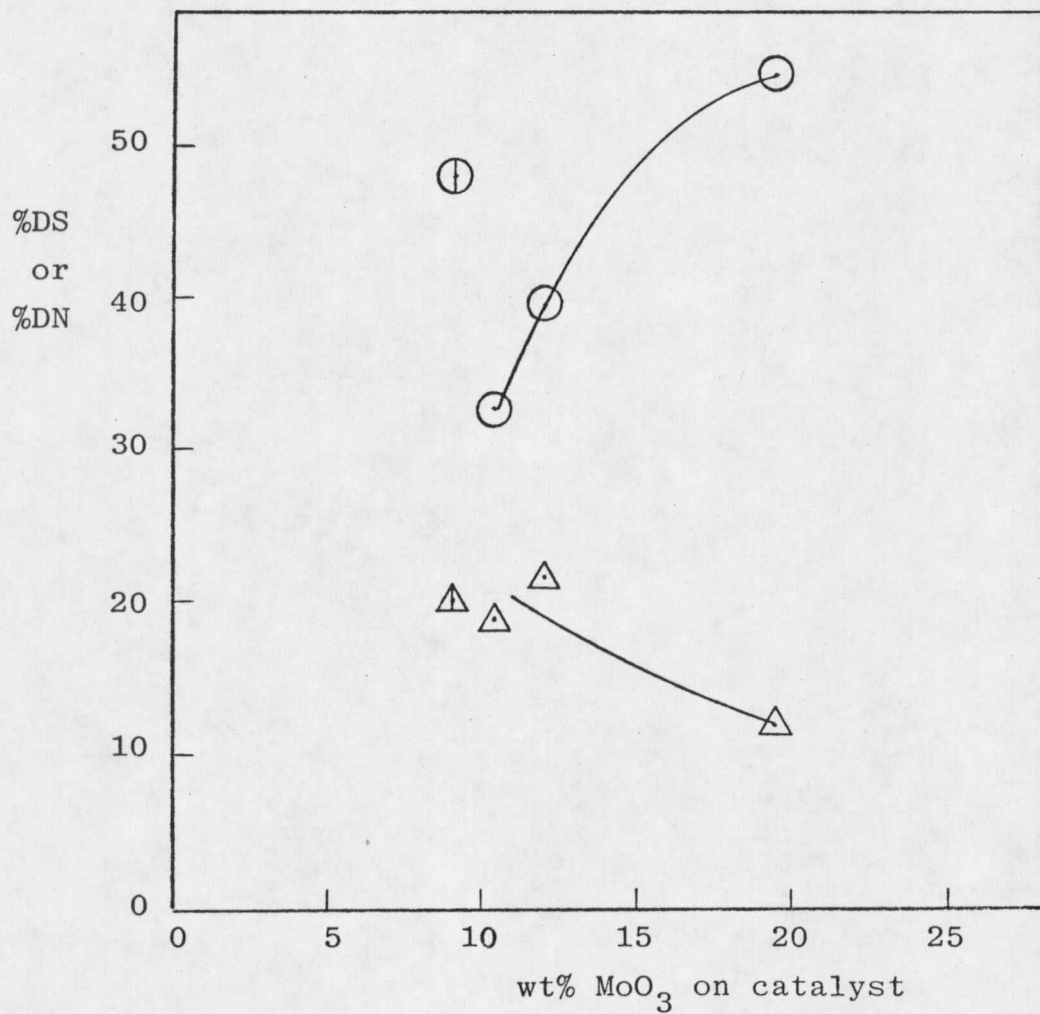


FIGURE 4. Effect of MoO₃ concentration on nitrogen and sulfur removals for base A, NALCO-78-6008A-1/32". The data is from batch runs.

○ DS
 △ DN
 ⊕ △ with additional WO₃

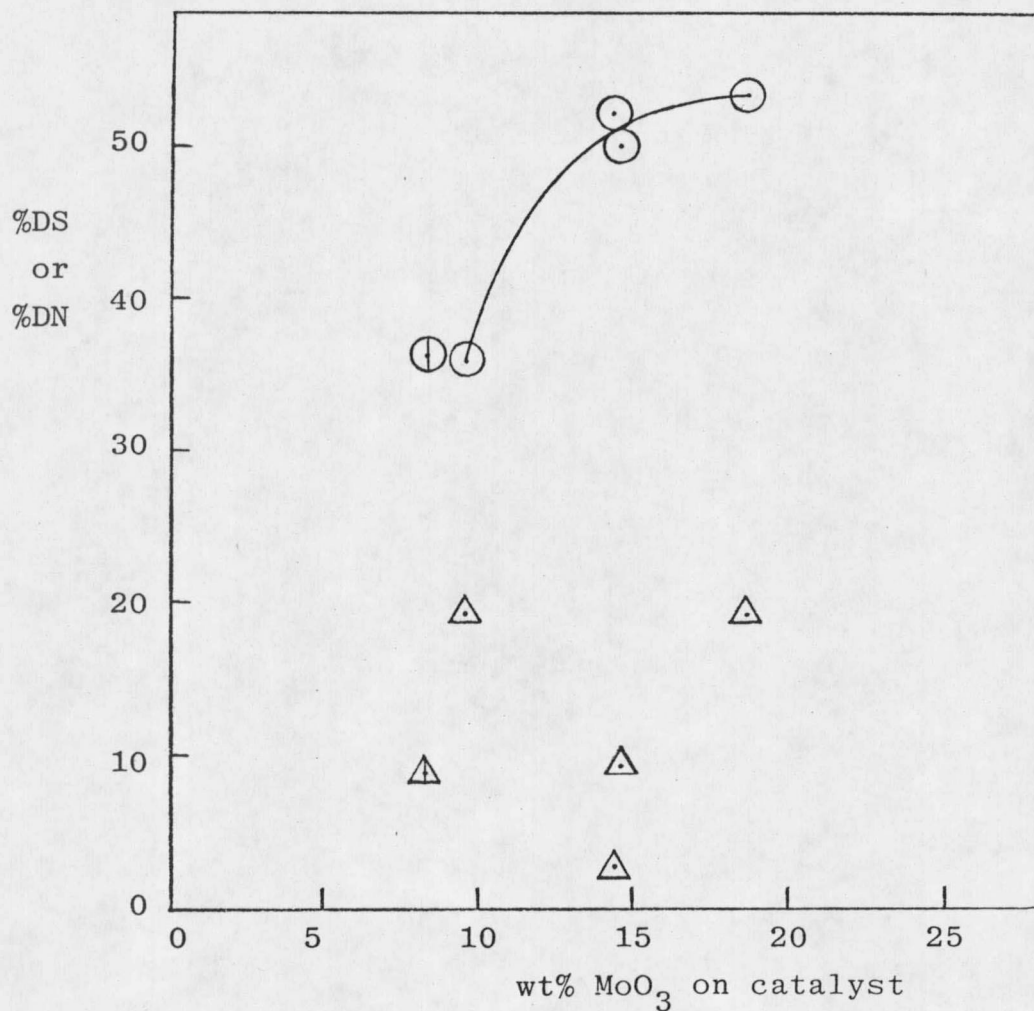


FIGURE 5. Effect of MoO_3 concentration on nitrogen and sulfur removals for base B, NALCO-78-6008B-1/16". The data is from batch runs.

○ DS
 △ DN
 ⊕ △ with additional WO_3

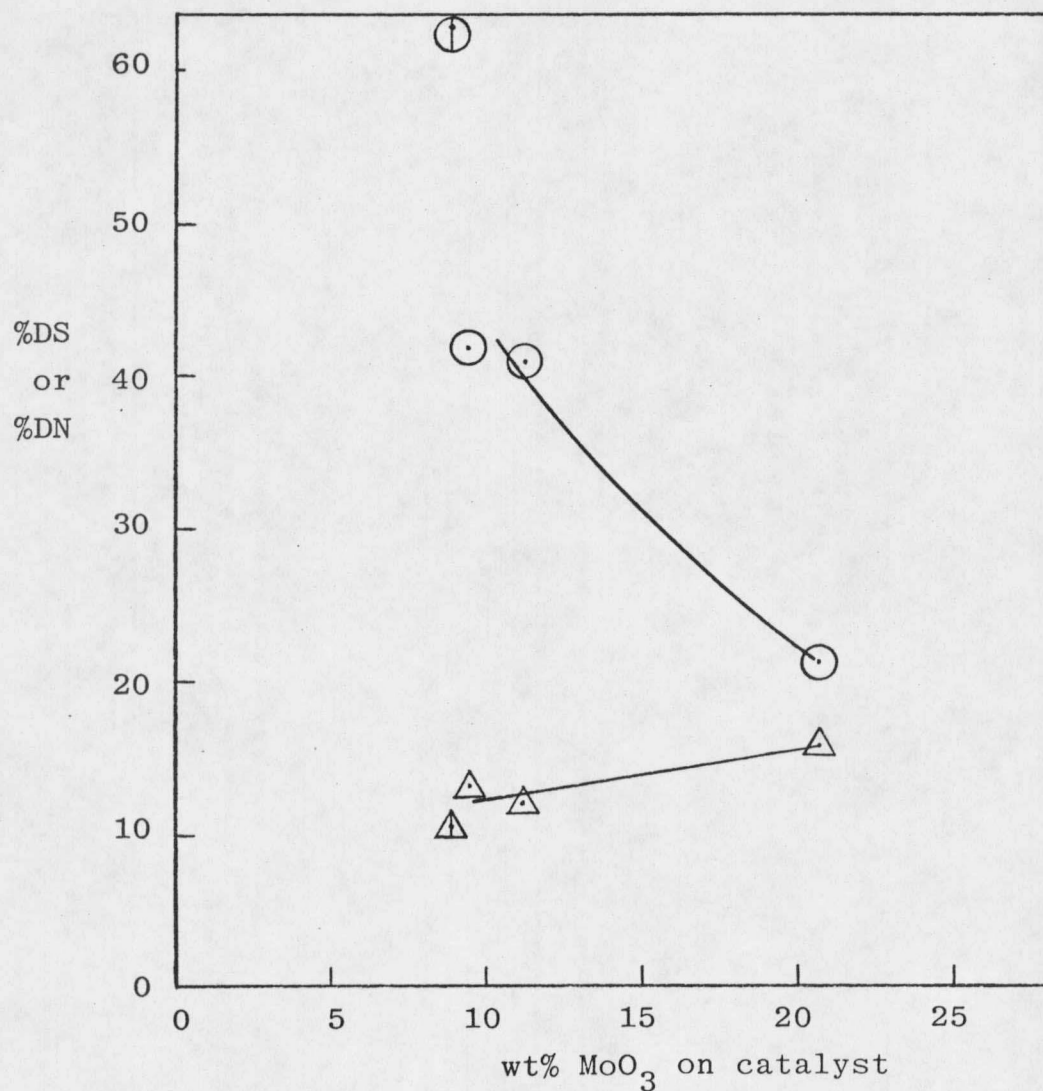


FIGURE 6. Effect of MoO_3 concentration on nitrogen and sulfur removals for the base C, NALCO-78-6008 C-1/32". The data is from batch runs.

○ DS
△ DN
⊕ △ with additional WO_3

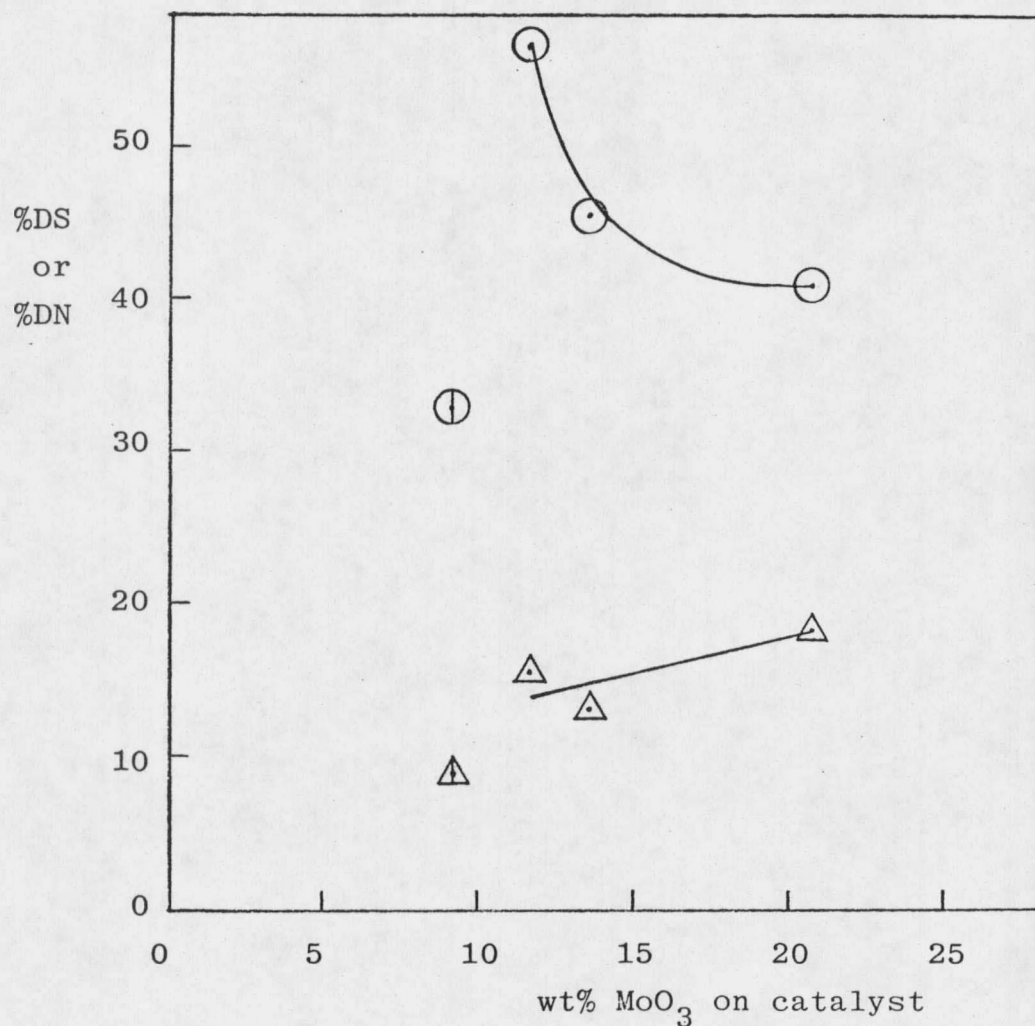


FIGURE 7. Effect of MoO_3 concentration on nitrogen and sulfur removals for the base D, NALCO 78-6008D-1/16". The data is from batch runs.

○ DS
△ DN
⊕ △ with additional WO_3

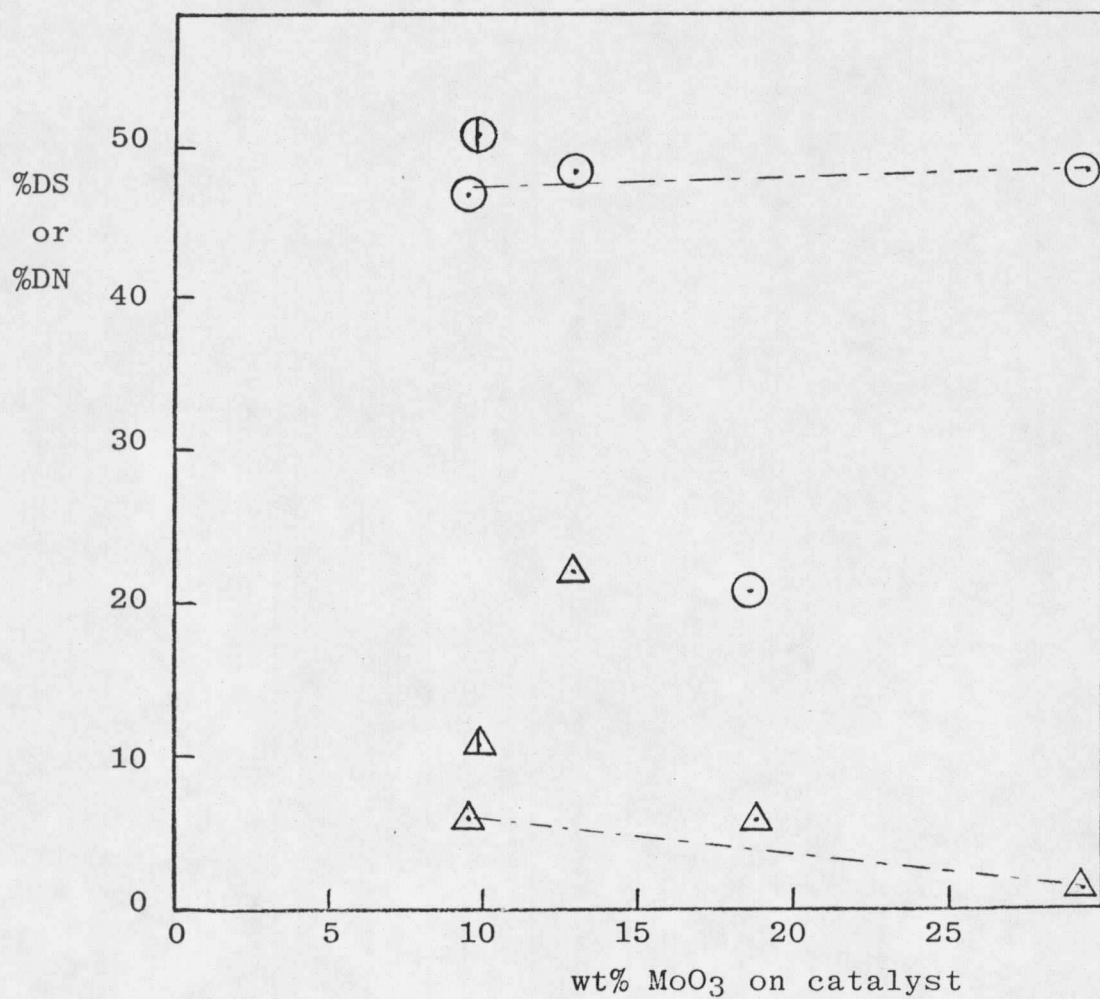


FIGURE 8. Effect of MoO₃ concentration on nitrogen and sulfur removals for base E, NALCO-78-6008E-1/16". The data is from batch runs.

○ DS
△ DN

⓪ ⓐ with additional WO₃

runs are so scattered that little information can be concluded from it.

Since only limited information can be obtained from batch runs, all catalysts were reexamined in a shorter time continuous run whenever possible.

Continuous Trickle Bed Reactor Runs

Catalysts were tested in the trickle bed reactor to determine the effects of operating conditions and the effects of specific chemical and physical catalyst properties on the hydrotreating of the SRC-II product. Continuous runs designated A-1 to A-25 were made to estimate the activity of 25 catalysts. The space velocity of these runs were kept at 1.0 hr^{-1} except that Run A-2 was at 0.5 hr^{-1} . The pressure was about 1,000 psig. Runs A-22 to A-25 were shut down when the pressure reached 1,300 psig. The detailed data of these runs are shown in Appendix B.

Runs A-1 to A-4 were 12 hours in length. Samples were taken every three hours as previously described. In an attempt to compare the result of Run A-1 with

previous work(23), the temperature was kept at 450 ± 5 °C. The upper zone of the catalyst bed was packed with Harshaw HT-400E-1/16", the lower zone with Cyanamid HDS-20A-1/16" Trilobe. Each was 30 ml. of catalyst diluted with 30 ml. of Denstone 1/8" inert support. It gave a better denitrogenation but it resulted in a pattern of catalyst deactivation very similar to the work done by Hass(23). This double catalyst zone showed no improvement in the catalyst life. Furthermore it makes the evaluation of each catalyst activity individually more difficult. It was then discontinued.

By reviewing previous work (23), it was found that almost all catalysts already investigated deactivate very rapidly on denitrogenation. They behave just like Run A-1 in Figure 9. Even though a catalyst shows high activity in initial period of running time, it is not necessarily the best from a commercial standpoint. Very active catalysts do not always have a long life and their expense has often led to the

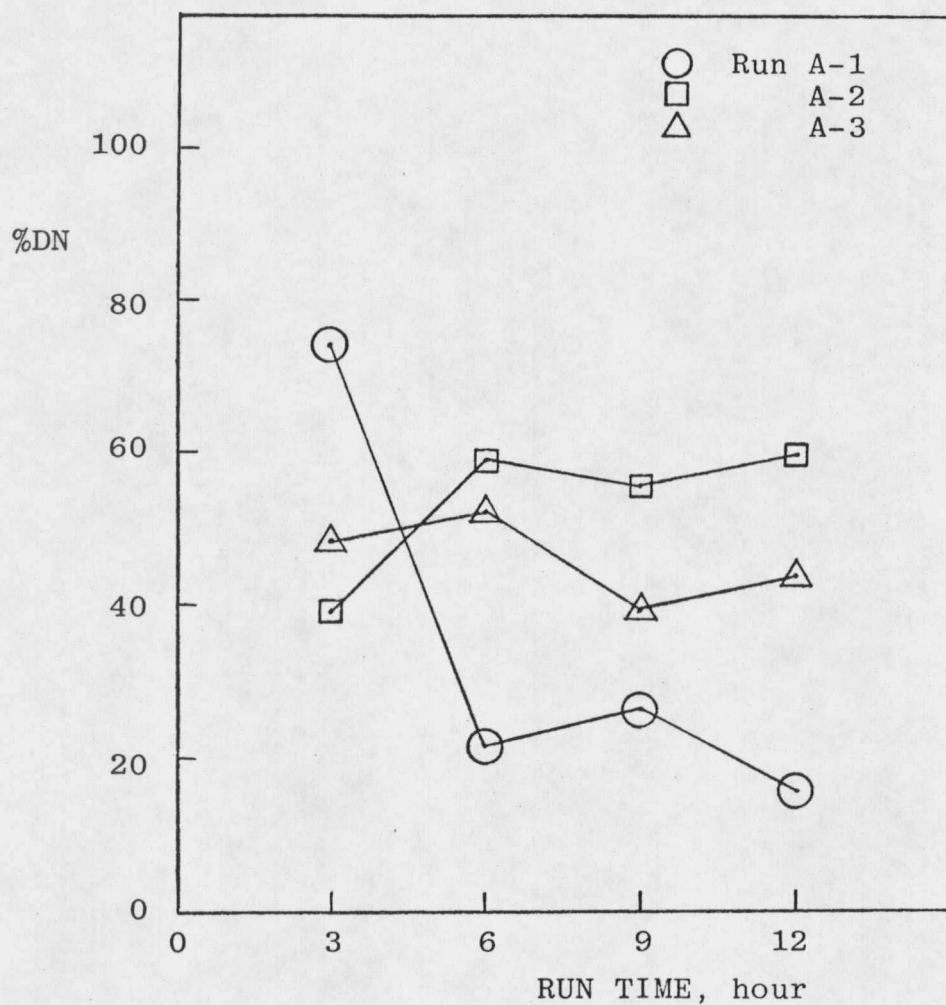


FIGURE 9. Effect of starting at a lower temperature on denitrogenation.

Run A-1 was kept constant temperature, 450°C , during the whole running period; Runs A-2 and A-3 were started at 335°C , then increased to 425°C within two hours.

use of less reactive but more durable catalysts in commercial operation. In an attempt to obtain a longer catalyst life on denitrogenation, Runs A-2 and A-3 were started at a lower temperature, 335 °C, then increased to 425±5 °C within two hours. Figures 9, 10, and 11 plot the results of these two runs on denitrogenation, desulfurization and distillation yield. By comparing the results of Runs A-2 and A-3 with A-1 in Figure 9, it can be seen that the activity of the catalyst is more durable and stable in denitrogenation by starting at a lower temperature. The comparison of distillation results in Figure 10 also shows a better behavior of catalyst. The activity of catalysts on desulfurization is compared in Figure 11. It points out that there is not much influence on desulfurization with this modified operating temperature.

The catalyst of Run A-4 was backflowed with preheated SRC-II product at 335 °C before starting up. This procedure was to prevent the SRC-II product from flowing in a non-ideal pattern in the reactor and to

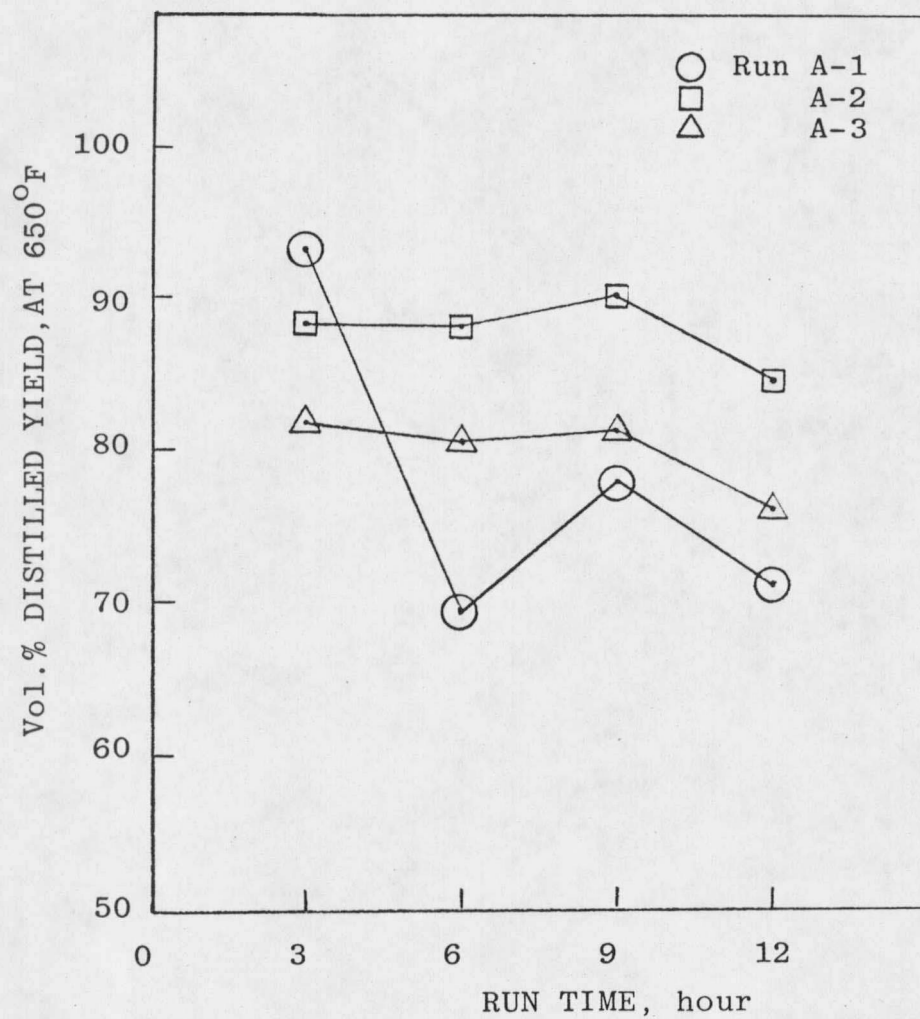


FIGURE 10. Effect of starting at a lower temperature on distillation yield at 650°F.

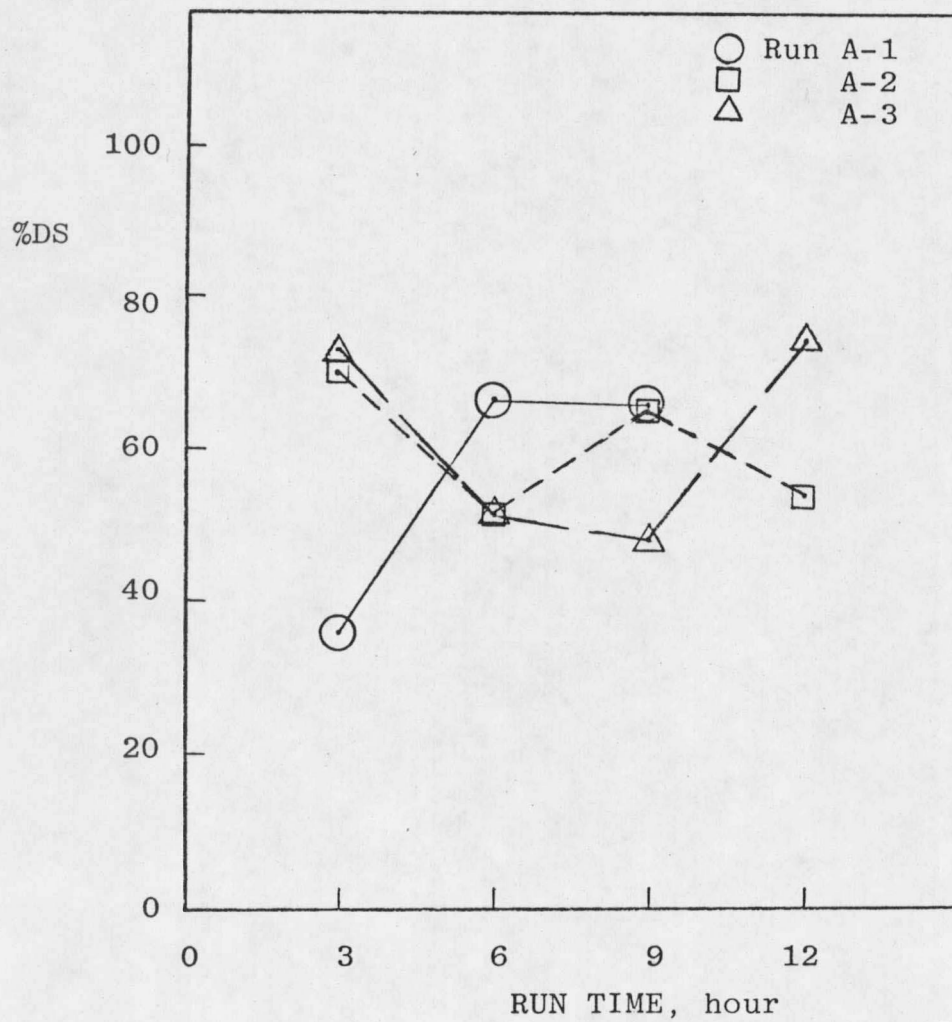


FIGURE 11. Effect of starting at a lower temperature on desulfurization.

allow the reactor to be fully wetted. However, the poor result of this run showed no help in preserving catalyst activity with this procedure. The data from Runs A-1 to A-4 is summarized in Table VIII.

Runs A-5 to A-25 were carried out for two hours or a little longer. Ramer(47) showed that the catalyst gave a good nitrogen removal for the first two hours. It was then decided to take samples as follows : one 30 minute sample, then six 15 minute samples, then 30 minute samples. As mentioned above sulfur analyses were done periodically on these samples, both because catalyst deactivation is much less rapid for sulfur removal and because the large number of samples in these runs would make it extremely difficult for the analysts to analyze all samples.

Runs A-5 and A-8 were started at 400 °C and heated to 425 °C in one hour and run for another 1.5 hours. Runs A-6 and A-7 were started at 350 °C and increased to 425 °C within 1.5 hours, then run for another one hour. The results of these runs showed

TABLE VIII
CONTINUOUS RUN DATA SUMMARY, A-1 to A-4

Run	Catalyst	Time, hours	wt%DS	wt%DN	%Yield*
A-1	Harshaw HT- 400E 1/16" and Cyanamid HDS -20A 1/16", Trilobe	3	36.8	67.2	93
		6	66.7	21.5	69.3
		9	65.9	25.8	77.6
		12	45.1	15.5	71.3
A-2	NALCO NM 502 1/16"	3	70.4	39.7	88.2
		6	51.4	60.3	88.0
		9	65.3	55.6	90.0
		12	54.2	59.0	84.6
A-3	NALCO MO 477 1/16"	3	72.9	48.3	82.2
		6	51.4	52.2	80.7
		9	48.6	39.2	81.3
		12	73.6	44.4	76.7
A-4	Harshaw HT- 400E 1/16" 720A-2-1-1	3	40.9	23.1	73.8
		6	70.1	38.0	74.6
		9	34.7	19.2	72.4
		12	58.3	25.2	74.3

* ASTM volume% yield at 650°F

that good nitrogen removal can be obtained during the first hour. The best was 89.6% denitrogenation (0.12 wt% nitrogen content) for Run A-7 at the end of the first hour.

In an attempt to investigate the effect of base composition and metal loading on the activity of the catalyst, Runs A-9 to A-20 were operated at the same conditions. The temperature was kept at 425 °C during the entire running period. The initial nitrogen removal was increased to as high as 96.6% (0.04 wt% nitrogen content) for some of these runs. A summary of nitrogen content, sulfur content, and distillation data for the first sample of each run is presented in Table IX. Figure 12 plots the activity of the catalyst for denitrogenation at the first 30 minute period for Runs A-9 to A-20. This plot points out that the major effect on denitrogenation is the catalyst base composition. It can be seen that the bases with the larger surface areas gave the better denitrogenation independently of the amount of MoO_3 loading. The best of

TABLE IX
INITIAL ACTIVITY OF CONTINUOUS
RUNS, A-5 to A-25

Run No.	Catalyst	wt%S [*]	wt%N ₂ ^{**}	IBP	ASTM-D86 ^{***} Vol.% distilled yield at 650°F
A-5	MSU-C20-E	.28	.26	337	81.0
A-6	MSU-C8-B	.25	.57	290	82.6
A-7	MSU-C5-B	.29	.19	329	83.8
A-8	MSU-C4-A	.20	.15	211	92.7
A-9	MSU-C3-A	.22	.04	308	86.9
A-10	MSU-C2-A	.20	.04	263	86.7
A-11	MSU-C1-A	.27	.08	242	89.8
A-12	MSU-C14-D	.24	.29	251	81.8
A-13	MSU-C12-C	.43	.39	320	73.6
A-14	MSU-C13-C	.17	.10	350	77
A-15	MSU-C23-B	.20	.13	236	87.4
A-16	MSU-C24-E	.19	.22	230	82.7
A-17	MSU-C9-B	.16	.14	181	87.2
A-18	MSU-C10-C	.26	.34	183	89.6
A-19	MSU-C15-D	.22	.35	253	86.2
A-20	MSU-C16-D	.18	.44	238	84.1
A-21	MSU-C27-C	.16	.06	171	93.9
A-22	MSU-C25-A	.27	.14	199	90.2
A-23	MSU-C29-E	.18	.05	192	84.2
A-24	MSU-C26-B	.20	.01	188	91.6
A-25	MSU-C28	.19	0	170	88.4

* First 30 minute or 45 minute sample

** First 30 minute sample

*** Cumulated 2 hour samples

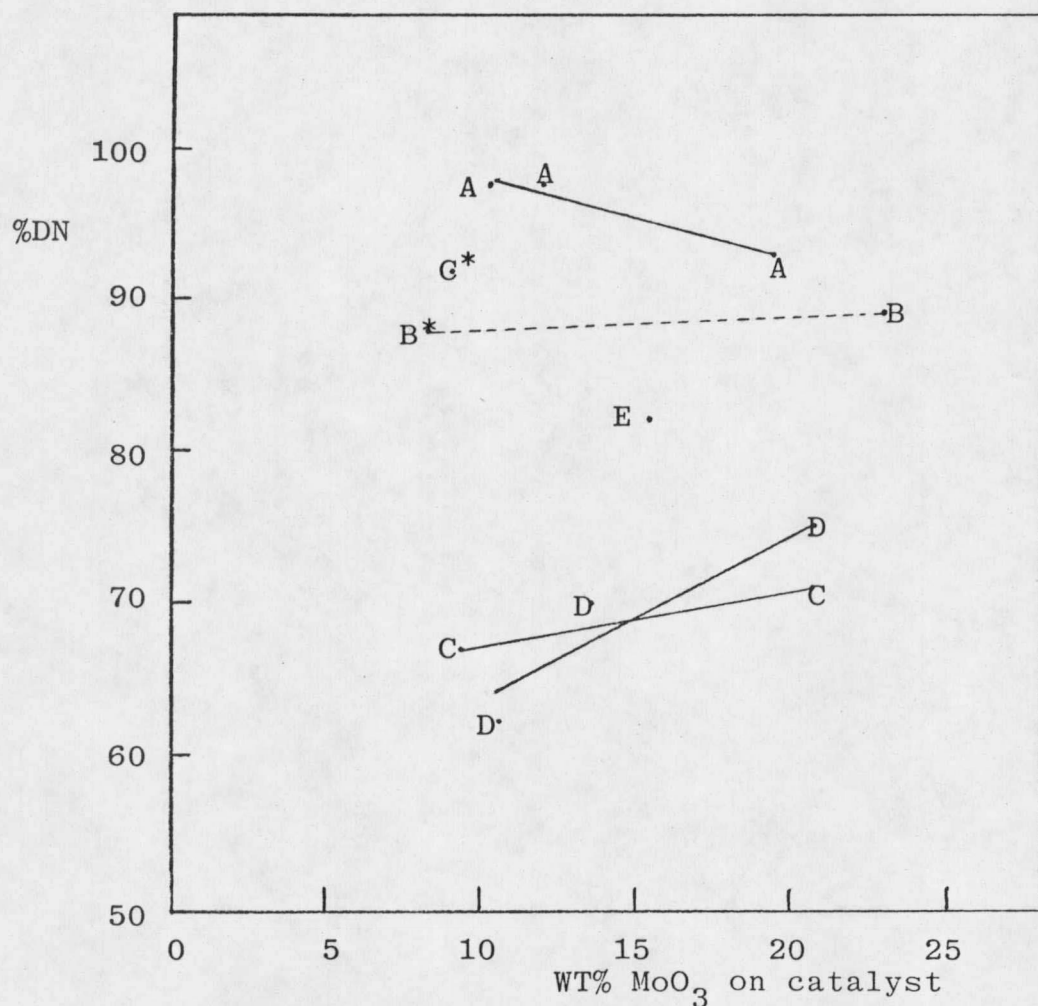


FIGURE 12. Effects of catalyst base and MoO₃ concentration on initial nitrogen removal, data is from Runs A-9 to A-20.

A : NALCO-78-6008A-1/32"; surface area, 323.2
 B : NALCO-78-6008B-1/16"; surface area, 232.43
 C : NALCO-78-6008C-1/32"; surface area, 214.57
 D : NALCO-78-6008D-1/16"; surface area, 211.39
 E : NALCO-78-6008E-1/16"; surface area, 146.95
 * : with additional WO₃

these bases is NALCO-78-6008A-1/32" which possesses the largest surface area, 323 m²/gm. NALCO-78-6008E-1/16" containing the smallest surface area but the largest median pore diameter, 420.4 Å, was not the poorest performer as might have been expected. It appears that the optimum base material is going to be a combination of proper surface area and pore diameter. The effect of MoO₃ content on denitrogenation, shown in Figure 12, is dependent on the catalyst used. This result is the same as that concluded from the batch data.

Runs A-21 to A-25 used the same operating conditions as previous runs except the length of the preheat zone. In these runs the preheat zone was packed with 200 ml. inert supports while it was 150 ml. inert supports prior to Run A-20. Ramer(48) showed that better results for denitrogenation were obtained by using a longer preheat section.

The data from Runs A-21 to A-25 showed that nitrogen removal was significantly improved during the first

three or four hours. However, the pressure drop in the packed bed went up by 250-300 psi very soon after three or four hours. Due to the limitation of equipment, Runs A-22 to A-25 were shutdown when the pressure reached 1,300 psig. Run A-21 was made for only two hours and no pressure drop was found. This increase in pressure drop during operation was found by Ramer(48). It is attributed to the carbon laydown on the reactor bed. It was found that most of carbon laydown is at the bottom of the preheat section before it comes into contact with the catalyst. Figures 13, 14, and 15 compare the denitrogenation, desulfurization and distillation results of Run A-21 with A-14. These runs used the same operating conditions, the same catalyst base, and similar metal composition, but had a different length of preheat section.

The catalysts used for Runs A-21 and A-14 were MSU-C27-C and MSU-C13-C respectively. Their metal composition are as follows :

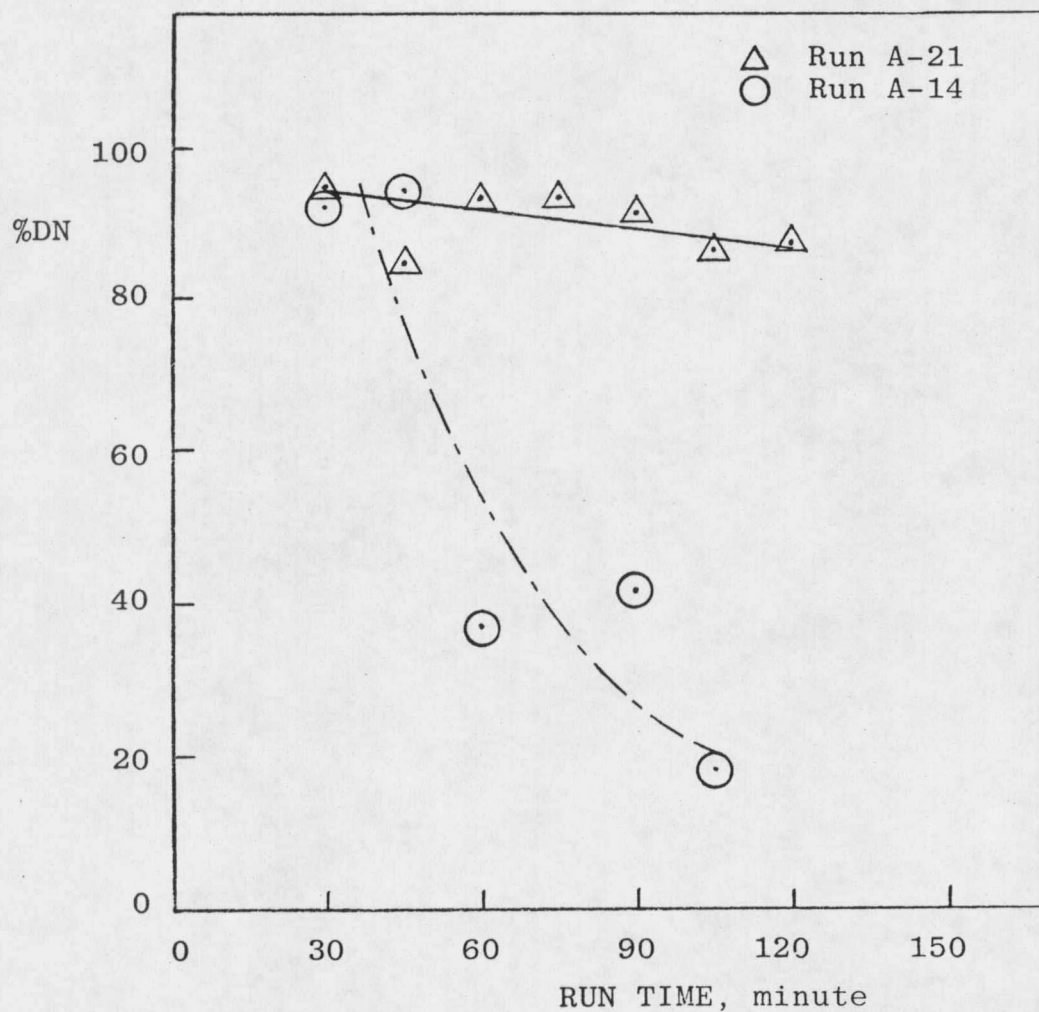


FIGURE 13. Different activity on denitrogenation for Runs A-21 and A-14.

Catalyst used :

A-21 : MSU-C27-C	11.5%MoO ₃	3.2%NiO	6.6%CoO
	9.5% WO ₃		
A-14 : MSU-C13-C	9.0%MoO ₃	.8%NiO	1.6%CoO
	9.0% WO ₃		

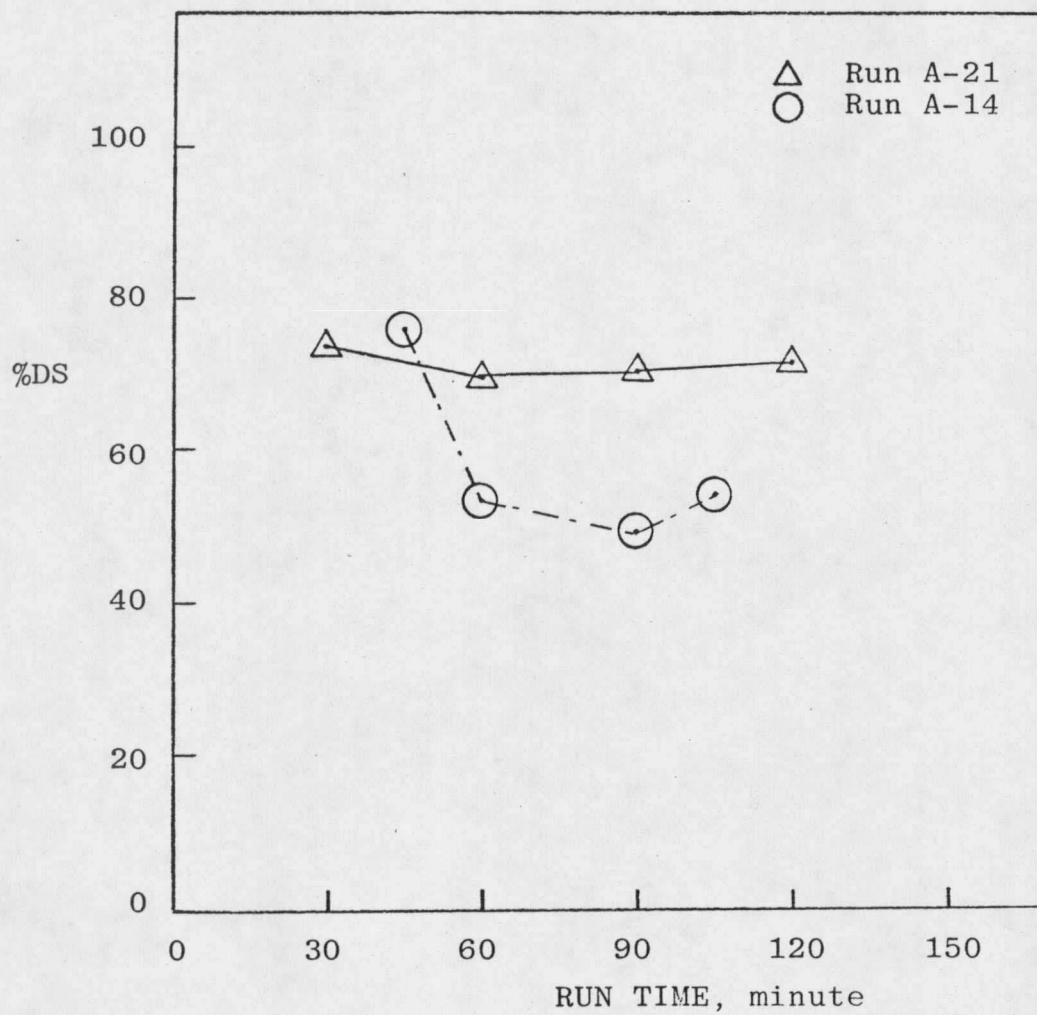


FIGURE 14. Different activity on desulfurization for Runs A-21 and A-14.

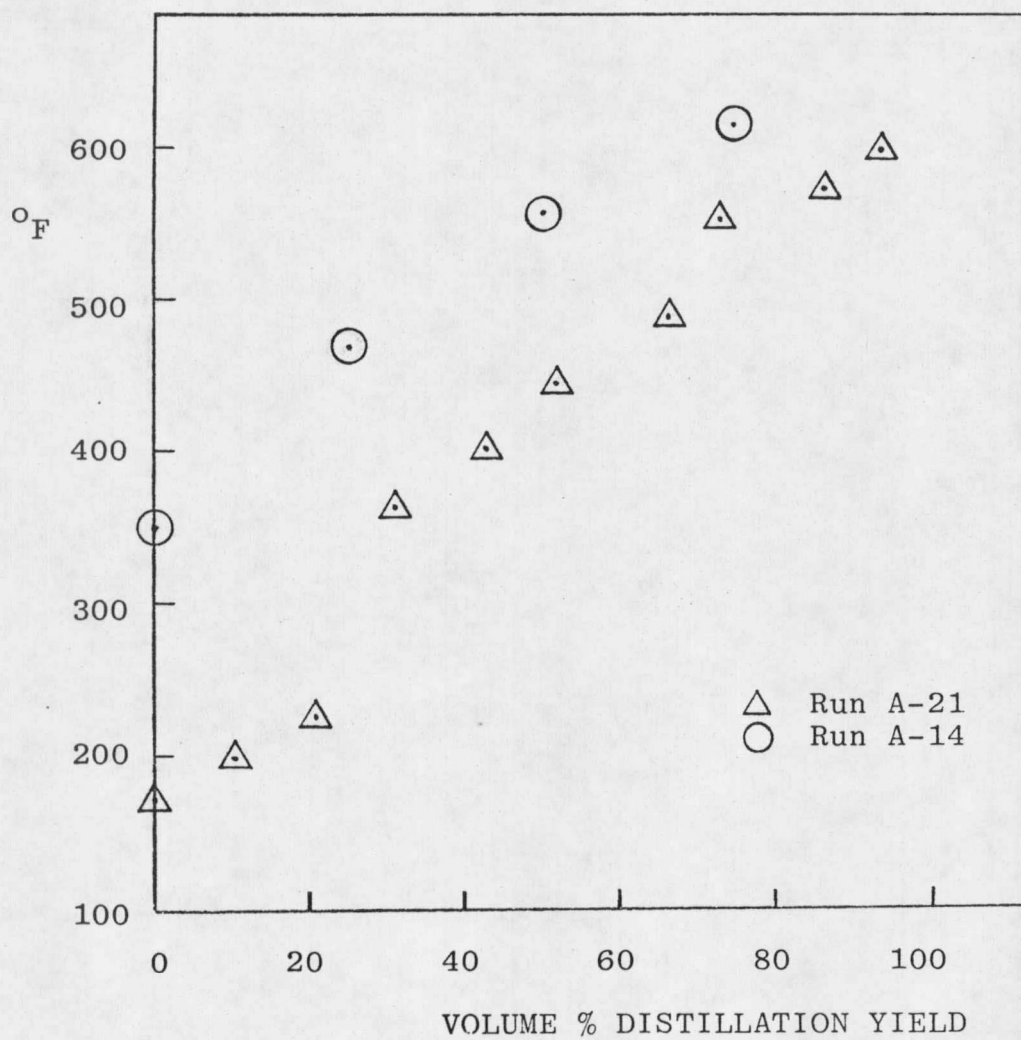


FIGURE 15. Different activity on distillation results for Runs A-21 and A-14.

A-21 : MSU-C27-C 11.54%MoO₃ 3.16%NiO 6.6%CoO .9%WO₃

A-14 : MSU-C13-C 9.05%MoO₃ 0.79%NiO 1.6%CoO 9.0%WO₃

It will be noted in Figure 13 that the activity of catalyst in Run A-21 is much better than Run A-14. The catalyst deactivation for denitrogenation in Run A-21 is much less rapid than in Run A-14. Two additional figures present the effect of the length of preheat section in denitrogenation. Figure 16 compares Run 18 with A-16 and Figure 17 compares Run 17 with A-13. Runs 17 and 18 were made by Ramer(48) using the longer preheat zone while Runs A-13 and A-16 used the shorter preheat zone made in this research. The operation conditions of these runs were otherwise the same. The catalysts used were fabricated by the author. They are as follows :

Run 18 : MSU-C21-E 9.5%MoO₃ 0.37%NiO 1.89%CoO

A-16 : MSU-C24-E 15.5%MoO₃ 0.15%NiO 2.30%CoO

Run 17 : MSU-C11-C 11.1%MoO₃ 0.10%NiO 1.60%CoO

A-13 : MSU-C12-C 9.5%MoO₃ 0.44%NiO 1.77%CoO

Although the catalysts used in these runs are not

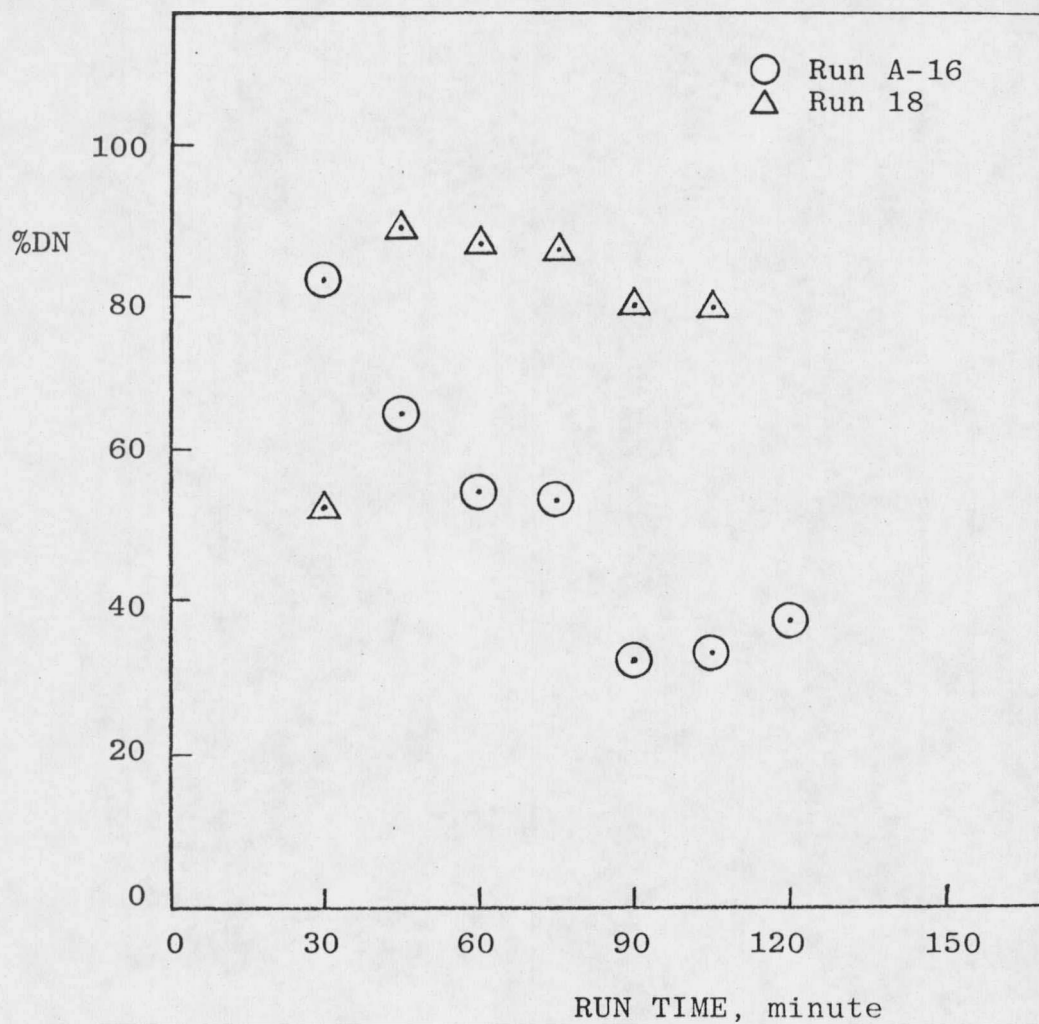


FIGURE 16. Different activity of catalyst for nitrogen removal by comparing Run A-16 with Run 18.

Catalyst used :

Run 18 : MSU-C21-E 9.5%MoO₃ .4%NiO 1.9%CoO

Run A-16 : MSU-C24-E 15.5%MoO₃ .2%NiO 2.3%CoO

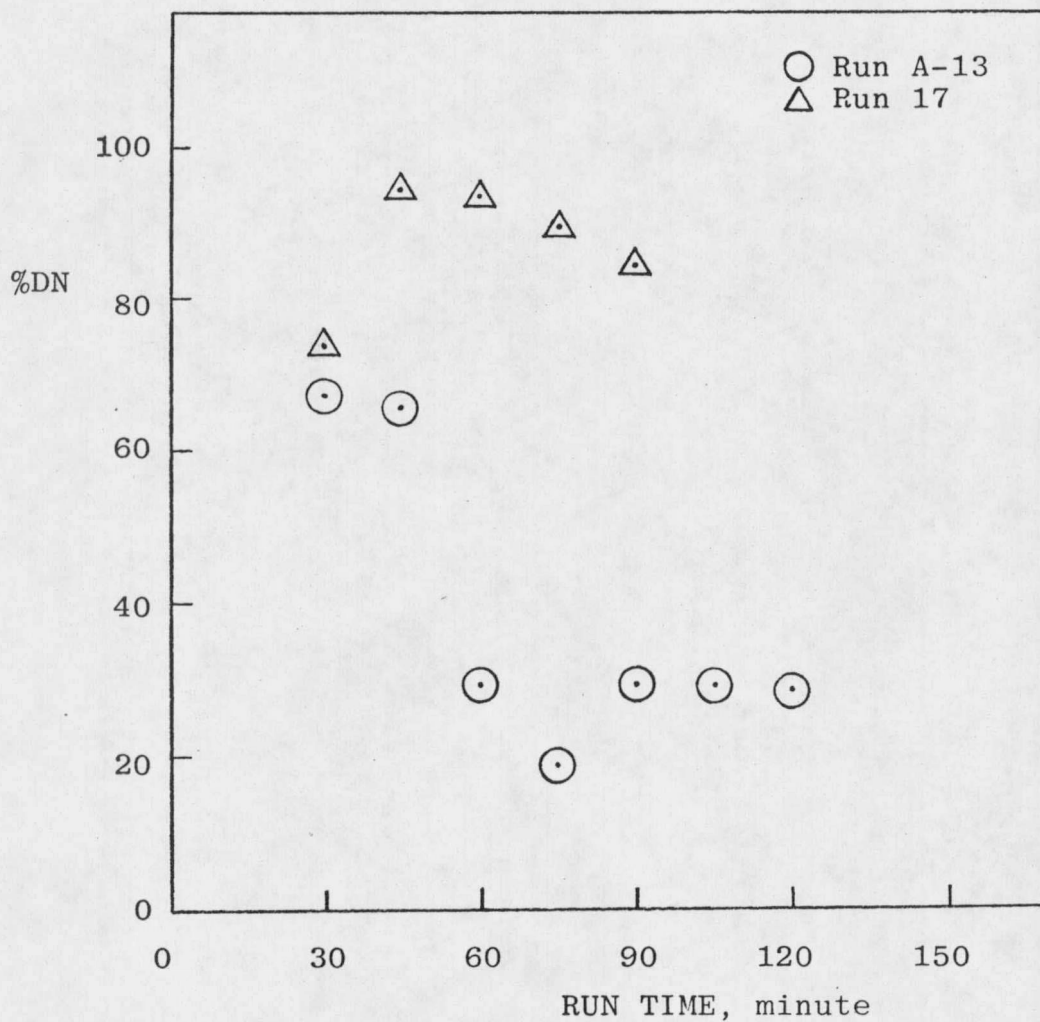


FIGURE 17. Different activity of catalyst for nitrogen removal by comparing Run A-13 with Run 17.

Catalyst used :

Run A-13 : MSU-C12-C 9.5%MoO₃ .4%NiO 1.8%CoO

Run 17 : MSU-C11-C 11.1%MoO₃ .1%NiO 1.6%CoO

exactly the same, it is believed that the length of preheat zone is the major factor in difference in nitrogen removal.

Nitrogen removal is approaching an acceptable level at this point. Since the carbon laydown on the packed bed is rapid, a burn-off process should be developed if the catalyst is to be reused. However, it is difficult at this time to determine which catalyst is the best and most feasible to be commercialized from the data of Runs A-21 to A-25. One of the criteria to evaluate the catalyst is the product yield. Figure 18 plots the effect of pore volume of the catalyst on the liquid product yields from Runs A-21 to A-25. It shows that the higher pore volume catalyst base gives the higher liquid product yield. NALCO-6008-78C-1/32" possessing the largest pore volume, 0.8397 c.c./gm, gave the highest liquid product yield of 82.9 vol.%. The best catalyst investigated in this research appears to be MSU-C27-C with the metal composition of 11.54%MoO₃ 6.6%CoO 3.16%NiO 9.5%WO₃ loaded on the base

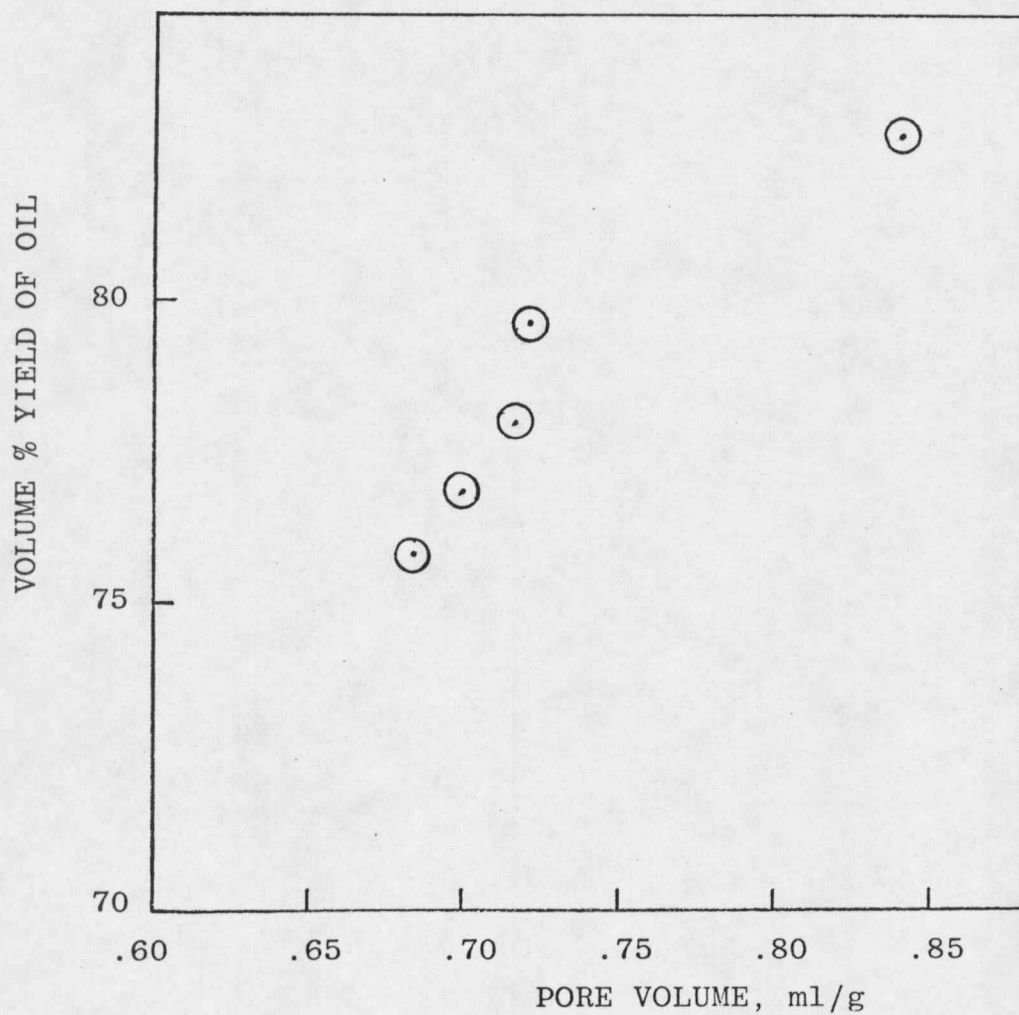


FIGURE 18. Effect of pore volume on liquid product yield, data is from Runs A-21 to A-25.

NALCO-6008-78C-1/32". The base possesses a surface area of $214.57 \text{ m}^2/\text{g}$, an average pore diameter of $156.5 \text{ \AA}$ and a pore volume of 0.8397 ml/g .

SUMMARY AND CONCLUSIONS

1. Catalyst deactivation is reduced by starting up at a lower temperature.
2. Doubling the catalyst bed did not give an improvement in catalyst deactivation and made catalyst evaluation more difficult.
3. The effect of MoO_3 concentration on nitrogen removal depends on the physical properties of the catalyst base used.
4. The higher surface area base gives better nitrogen removal.
5. A high pore diameter base showed an improvement on the nitrogen removal. The catalyst base with an optimum combination of proper surface area and pore diameter appeared to be important.
6. The effect of catalyst base properties is more significant than MoO_3 concentration on the nitrogen removal.
7. A satisfactory nitrogen and sulfur removal was obtained by the packed bed with the longer preheat zone, in which the catalyst lasted for three to four

hours before carbon laydown forced the shutdown

8. In the reactor with the longer preheat zone, the pressure drop increased by 250 psi after three hours operation which was caused by carbon laid down in the preheat section.
9. The larger pore volume catalyst base gave the higher yield of liquid product.

RECOMMENDATION FOR FUTURE RESEARCH

1. Batch runs do not give enough information; all catalysts should be tested in continuous runs.
2. The optimum combination of proper surface area and pore diameter on a base material should be determined.
3. A burn-off process should be developed to retest the used catalyst in a reactor packed with the longer preheat section.
4. A lower liquid feed rate might be used to reduce the pressure drop.
5. The 1/8 inches pellet inert support might be replaced by the same size spherical inert support to reduce the pressure drop.
6. A recycle process should be developed to determine if pressure drop will be reduced.
7. Chromatographic analysis of the exit gases should be performed so that a material balance may be made on the reactor.

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APPENDIX A
BATCH RUN DATA

Run No. B-1

Catalyst No. MSU-C1-A

Catalyst Composition

Metals : 19.4%MoO₃ .1%NiO .43%CoO

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2
 Base Pore Volume, ml/g : .7183
 Base Pore Diameter (4V/A), Å : 88.9
 Base Median Pore Diameter, Å : 90.2

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	380
At Run Temperature :	3,900	1,800

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:105 Residence Time, min:75

Maximum Pressure, psig: 4,300

Weight % Desulfurization: 55.6

Weight % Denitrogenation: 12.0

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 34.8 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	341	405	440	482	537	581	614	642

Run No. B-2

Catalyst No. MSU-C2-A

Catalyst Composition

Metals : 12.0%MoO₃ .48%NiO .1%CoO

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2
 Base Pore Volume, ml/g : .7183
 Base Pore Diameter (4V/A), Å : 88.9
 Base Median Pore Diameter, Å : 90.2

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	480
At Run Temperature :	4,300	2,000

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:120 Residence Time, min:60

Maximum Pressure, psig: 4,360

Weight % Desulfurization: 39.6

Weight % Denitrogenation: 21.4

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.7 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 306	384	440	487	535	578	624	667

Run No. B-3

Catalyst No. MSU-C3-A

Catalyst Composition

Metals : 10.4%MoO₃ 1.16%NiO .11%CoO

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2
 Base Pore Volume, ml/g : .7183
 Base Pore Diameter (4V/A), Å : 88.9
 Base Median Pore Diameter, Å : 90.2

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,490	900
At Run Temperature :	3,800	2,800

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min: 75 Residence Time, min: 105

Maximum Pressure, psig: 4,400

Weight % Desulfurization: 32.6

Weight % Denitrogenation: 18.8

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.6 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F :	309	405	457	501	541	592	649	672

Run No. B-4

Catalyst No. MSU-C4-A

Catalyst Composition

Metals : 9.1%MoO₃ .37%NiO 1.38%CoO 8.2%WO₃

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2
 Base Pore Volume, ml/g : .7183
 Base Pore Diameter (4V/A), Å : 88.9
 Base Median Pore Diameter, Å : 90.2

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	550
At Run Temperature :	4,150	2,100

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min: 105 Residence Time, min: 75

Maximum Pressure, psig: 4,550

Weight % Desulfurization: 47.9

Weight % Denitrogenation: 20.1

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 38.1 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F :	316	399	445	489	541	587	620	654

Run No. B-5

Catalyst No. MSU-C5-B

Catalyst Composition

Metals : 18.6%MoO₃ .26%NiO .45%CoO

Base : NALCO-78-6008B-1/16"

Base Surface Area, m²/g : 232.4

Base Pore Volume, ml/g : .7215

Base Pore Diameter (4V/A), Å : 124.2

Base Median Pore Diameter, Å : 137.3

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	400
At Run Temperature :	4,020	1,900

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:120 Residence Time, min:60

Maximum Pressure, psig: 4,400

Weight % Desulfurization: 52.8

Weight % Denitrogenation: 19.2

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.95 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	331	409	436	459	504	561	626	649

Run No. B-6

Catalyst No. MSU C6-B

Catalyst Composition

Metals : 14.8%MoO₃ .19%NiO 1.1%CoO

Base : NALCO-78-6008B-1/16"

Base Surface Area, m ² /g	:	232.4
Base Pore Volume, ml/g	:	.7215
Base Pore Diameter (4V/A), Å	:	124.2
Base Median Pore Diameter, Å	:	137.3

Feed Charge:	SRC-II Product: 200 g
	Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,430	430
At Run Temperature :	4,190	2,000

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min: 105 Residence Time, min: 75

Maximum Pressure, psig: 4,250

Weight % Desulfurization: 50.0

Weight % Denitrogenation: 9.4

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 38.0 ml

Volume % :	IBP	10	20	30	40	50	60	70	80
°F :	342	420	459	510	550	600	642	661	

Run No. B-7

Catalyst No. MSU-C7-B

Catalyst Composition

Metals : 14.6%MoO₃ .13%NiO 5.8%CoO

Base : NALCO-78-6008B-1/16"

Base Surface Area, m²/g : 232.4
 Base Pore Volume, ml/g : .7215
 Base Pore Diameter (4V/A), Å : 124.2
 Base Median Pore Diameter, Å : 137.3

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	580
At Run Temperature :	4,300	2,220

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:120 Residence Time, min: 60

Maximum Pressure, psig: 4,400

Weight % Desulfurization: 52.1

Weight % Denitrogenation: 2.6

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 38.0 ml

Volume % :	IBP	10	20	30	40	50	60	70	80
°F :	341	410	451	490	549	582	622	637	

Run No. B-8

Catalyst No. MSU-C8-B

Catalyst Composition

Metals : 9.8%MoO₃ .45%NiO .43%CoO

Base : NALCO-78-6008B-1/16"

Base Surface Area, m ² /g	:	232.4
Base Pore Volume, ml/g	:	.7215
Base Pore Diameter (4V/A), Å	:	137.3
Base Median Pore Diameter, Å	:	124.2

Feed Charge:	SRC-II Product:	200 g
	Catalyst	: 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	420
At Run Temperature :	4,300	1,950

Run Temperature, °C : Target: 425 Range: ± 5

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

Maximum Pressure, psig: 4,300

Weight % Desulfurization: 36.1

Weight % Denitrogenation: 19.2

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.5 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 284	389	453	499	541	591	643	686

Run No. B-9

Catalyst No. MSU-C9-B

Catalyst Composition

Metals : 8.3%MoO₃ .57%NiO 1.55%CoO 8.8%WO₃

Base : NALCO-78-6008B-1/16"

Base Surface Area, m²/g : 232.4

Base Pore Volume, ml/g : .7215

Base Pore Diameter (4V/A), $\frac{Q}{A}$: 137.3

Base Median Pore Diameter, $\frac{Q}{A}$: 124.2

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,550	550
At Run Temperature :	4,200	2,050

Run Temperature, °C : Target: 425 Range: $\pm$ 2

Time At Run Temperature, min: 105 Residence Time, min: 75

Maximum Pressure, psig: 4,550

Weight % Desulfurization: 36.8

Weight % Denitrogenation: 8.5

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 36.7 ml

Volume % : IBP	10	20	30	40	50	60	70	80
$^{\circ}\text{F}$:	287	396	446	524	574	598	629	670

Run No. B-10

Catalyst No. MSU-C10-C

Catalyst Composition

Metals : 20.6%MoO₃ .1%NiO 1.08%CoO

Base : NALCO-78-6008C-1/32"

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,520	550
At Run Temperature :	3,600	2,450

Run Temperature, °C : Target:425 Range: ± 10

Time At Run Temperature, min:60 Residence Time, min:120

Maximum Pressure, psig: 4,480

Weight % Desulfurization:20.8

Weight % Denitrogenation:15.4

ASTM Distillation

Volume of Charge: 51 ml Final Volume: 39 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 241	327	417	464	498	551	592	647 670

Run No. B-11

Catalyst No. MSU-C11-C

Catalyst Composition

Metals : 11.1%MoO₃ .1%NiO 1.56%CoO

Base : NALCO-78-6008C-1/32"

Base Surface Area, m²/g : 214.57
 Base Pore Volume, ml/g : .8397
 Base Pore Diameter (4V/A), Å : 156.5
 Base Median Pore Diameter, Å : 161

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,350	680
At Run Temperature :	4,000	2,350

Run Temperature, °C : Target: 425 Range: ± 10

Time At Run Temperature, min:105 Residence Time, min: 75

Maximum Pressure, psig: 4,200

Weight % Desulfurization: 41.0

Weight % Denitrogenation: 12.0

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 38.5 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 291	401	448	492	539	591	634	658

Run No. B-12

Catalyst No. MSU-C12-C

Catalyst Composition

Metals : 9.5%MoO₃ .44%NiO 1.77%CoO

Base : NALCO-78-6008C-1/32"

Base Surface Area, m²/g : 214.57
 Base Pore Volume, ml/g : .8397
 Base Pore Diameter (4V/A), Å : 156.5
 Base Median Pore Diameter, Å : 161

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	630
At Run Temperature :	4,250	2,300

Run Temperature, °C : Target: 425 Range: ± 7

Time At Run Temperature, min:105 Residence Time, min:75

Maximum Pressure, psig: 4,380

Weight % Desulfurization: 41.7

Weight % Denitrogenation: 12.8

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 38.9 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 310	415	444	487	536	578	635	659

Run No.B-13

Catalyst No. MSU-C13-C

Catalyst Composition

Metals : 9.05%MoO₃ .79%NiO 1.6%CoO 9.0%WO₃

Base : NALCO-78-6008C-1/32"

Base Surface Area, m²/g : 214.57
 Base Pore Volume, ml/g : .8397
 Base Pore Diameter (4V/A), Å : 156.5
 Base Median Pore Diameter, Å : 161

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,570	700
At Run Temperature :	4,430	2,350

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:105 Residence Time, min: 75

Maximum Pressure, psig: 4,450

Weight % Desulfurization: 62.6

Weight % Denitrogenation: 10.3

ASTM Distillation

Volume of Charge: 50 ml

Final Volume: 38.8 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F : 265	394	440	482	531	580	619	638	

Run No. B-14

Catalyst No. MSU-C14-D

Catalyst Composition

Metals : 20.7%MoO₃ 1.86%NiO 1.2%CoO

Base : NALCO-78-6008D-1/16"

Base Surface Area, m²/g : 211.39
 Base Pore Volume, ml/g : .7943
 Base Pore Diameter (4V/A), $\frac{Q}{A}$: 150.3
 Base Median Pore Diameter, $\frac{Q}{A}$: 190

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,490	650
At Run Temperature :	4,010	2,250

Run Temperature, °C : Target: 425 Range: $\pm$ 5

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

Maximum Pressure, psig:4,220

Weight % Desulfurization: 41.0

Weight % Denitrogenation: 15.4

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.6 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F :	312	420	462	510	548	591	648	701

Run No. B-15

Catalyst No. MSU-C15-D

Catalyst Composition

Metals : 13.7%MoO₃ .78%NiO .1%CoO

Base : NALCO-78-6008D-1/16"

Base Surface Area, m²/g : 211.39

Base Pore Volume, ml/g : .7943

Base Pore Diameter (4V/A), Å : 150.3

Base Median Pore Diameter, Å : 190

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	800
At Run Temperature :	4,470	2,550

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:105 Residence Time, min:75

Maximum Pressure, psig:4,500

Weight % Desulfurization: 45.1

Weight % Denitrogenation: 12.8

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 40.7 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F :	324	397	432	468	510	564	606	617

Run No. B-16

Catalyst No. MSU-C16-D

Catalyst Composition

Metals : 11.8%MoO₃ .1%NiO 1.9%CoO

Base : NALCO-78-6008D-1/16"

Base Surface Area, m²/g : 211.39

Base Pore Volume, ml/g : .7943

Base Pore Diameter (4V/A), Å : 150.3

Base Median Pore Diameter, Å : 190

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,420	870
At Run Temperature :	4,020	2,600

Run Temperature, °C : Target:425 Range: ±12

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

Maximum Pressure, psig: 4,250

Weight % Desulfurization: 56.9

Weight % Denitrogenation: 15.4

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.0 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	306	416	459	508	554	596	647	720

Run No. B-17

Catalyst No. MSU-C17-D

Catalyst Composition

Metals : 9.3%MoO₃ .17%NiO 1.76%CoO 8.8%WO₃

Base : NALCO-78-6008D-1/16"

Base Surface Area, m²/g : 211.39

Base Pore Volume, ml/g : .7943

Base Pore Diameter (4V/A), Å : 150.3

Base Median Pore Diameter, Å : 190

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,600	900
At Run Temperature :	4,600	2,750

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:105 Residence Time, min: 75

Maximum Pressure, psig: 4,630

Weight % Desulfurization: 32.6

Weight % Denitrogenation: 8.5

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.8 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 291	398	446	480	527	579	611	629

Run No. B-18

Catalyst No. MSU-C18-E

Catalyst Composition

Metals : 29.3%MoO₃ .1%NiO .1%CoO

Base : NALCO-78-6008E-1/16"

Base Surface Area, m²/g : 146.95

Base Pore Volume, ml/g : .6841

Base Pore Diameter (4V/A), $\frac{Q}{A}$: 186.2

Base Median Pore Diameter, $\frac{Q}{A}$: 420.2

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,450	920
At Run Temperature :	4,100	2,750

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min:105 Residence Time, min: 75

Maximum Pressure, psig: 4,400

Weight % Desulfurization: 47.9

Weight % Denitrogenation: 1.7

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 37.9 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F : 315	421	445	512	573	604	622	681	

Run No. B-19

Catalyst No. MSU-C19-E

Catalyst Composition

Metals : 18.9%MoO₃ .1%NiO 3.5%CoO

Base : NALCO-78-6008E-1/16"

Base Surface Area, m ² /g	:	146.95
Base Pore Volume, ml/g	:	.6841
Base Pore Diameter (4V/A), Å	:	420.2
Base Median Pore Diameter, Å	:	186.2

Feed Charge:	SRC-II Product:	200 g
	Catalyst	25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,370	850
At Run Temperature :	4,000	2,700

Run Temperature, °C : Target: 425 Range: ± 5

Time At Run Temperature, min: 75 Residence Time, min: 105

Maximum Pressure, psig: 4,310

Weight % Desulfurization: 20.1

Weight % Denitrogenation: 6.0

ASTM Distillation

Volume of Charge:	50 ml	Final Volume:	35.6 ml
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Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 349	413	460	500	549	595	635	670

Run No. B-20

Catalyst No. MSU-C20-E

Catalyst Composition

Metals : 13.0%MoO₃ .1%NiO .3%CoO

Base : NALCO-78-6008E-1/16"

Base Surface Area, m ² /g	:	146.95
Base Pore Volume, ml/g	:	.6841
Base Pore Diameter (4V/A), Å	:	186.2
Base Median Pore Diameter, Å	:	420.2

Feed Charge:	SRC-II Product:	200 g
	Catalyst	: 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	700
At Run Temperature :	3,820	2,300

Run Temperature, °C : Target: 425 Range: ± 2

Time At Run Temperature, min:105 Residence Time, min:75

Maximum Pressure, psig: 4,370

Weight % Desulfurization: 47.9

Weight % Denitrogenation: 22.2

ASTM Distillation

Volume of Charge: 50.5 ml Final Volume: 38.2 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	: 327	428	461	512	557	601	651	691

Run No. B-21

Catalyst No. MSU-C21-E

Catalyst Composition

Metals : 9.5%MoO₃ .37%NiO 1.89%CoO

Base : NALCO-78-6008E-1/16"

Base Surface Area, m ² /g	:	146.95
Base Pore Volume, ml/g	:	.6841
Base Pore Diameter (4V/A), Å	:	186.2
Base Median Pore Diameter, Å	:	420.2

Feed Charge: SRC-II Product: 200 g
Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	900
At Run Temperature :	4,200	2,800

Run Temperature, °C : Target: 425 Range: ± 3

Time At Run Temperature, min: 105 Residence Time, min: 75

Maximum Pressure, psig: 4,500

Weight % Desulfurization: 46.0

Weight % Denitrogenation: 6.5

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 38.5 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F : 300	403	455	503	541	594	618	642	

Run No. B-22

Catalyst No. MSU-C22-E

Catalyst Composition

Metals : 9.8%MoO₃ .3%NiO 1.4%CoO 9.2%WO₃

Base : NALCO-78-6008E-1/16"

Base Surface Area, m²/g : 146.95
 Base Pore Volume, ml/g : .6841
 Base Pore Diameter (4V/A), Å : 186.2
 Base Median Pore Diameter, Å : 420.2

Feed Charge: SRC-II Product: 200 g
 Catalyst : 25 ml

Hydrogen Pressure, psig:

	<u>Initial</u>	<u>Final</u>
At Room Temperature:	2,500	900
At Run Temperature :	4,200	2,700

Run Temperature, °C : Target: 425 Range: $\pm$ 2

Time At Run Temperature, min:105 Residence Time, min: 75

Maximum Pressure, psig: 4,500

Weight % Desulfurization:51.5

Weight % Denitrogenation:11.1

ASTM Distillation

Volume of Charge: 52 ml Final Volume: 38.0 ml

Volume % : IBP	10	20	30	40	50	60	70	80
°F	302	419	468	501	549		660	

APPENDIX B
CONTINUOUS RUN DATA

Run No. A-1

Catalyst No. (1) Harshaw HT-400E 1/16"

(2) Cyanamid HDS-20A 1/16"

Metals : (1) 15%MoO₃ 3%CoO; (2) 16.2%MoO₃ 5%CoO

Surface Area, m²/g : (1) 220; (2) 230

Pore Volume, ml/g : (1) .5 ; (2) .52

Pore Diameter, (4V/A), Å: (1) 91 ; (2) 90

Run Temperature, °C : 450±5

Run Pressure, psig : 1,030

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, Hours:	3	6	9	12
wt% N ₂	: .38	.91	.86	.98
wt% S	: .46	.24	.24	.40

ASTM Distillation

3 hours Volume of Charge: 50 ml Final Volume: 46.5 ml

Volume: IBP	5	10	15	20	25	30	35	40	45
°F	: 185	300	380	416	440	478	530	568	600 630

6 hours Volume of Charge: 45 ml Final Volume: 39.5 ml

Volume: IBP	5	10	15	20	25	30	35	38
°F	: 210	340	423	466	512	567	608	653 684

(Run A-1 continued)

9 hours Volume of Charge: 50 ml Final Volume: 44.3 ml

Volume: IBP	5	10	15	20	25	30	35	40	43
°F :	206	338	418	456	490	532	570	608	663 678

12 hours Volume of Charge: 51 ml Final Volume: 41.9 ml

Volume: IBP	5	10	15	20	25	30	35	40
°F :	340	389	443	484	523	560	603	643 691

Run No. A-2

Catalyst No. NALCO NM-502 1/16"

Metals : 14.0%MoO₃ 4.0%CoO

Surface Area, m²/g : 240

Pore Volume, ml/g : .53

Pore Diameter, (4V/A), Å: 88

Run Temperature, °C : 425₊₅; started at 335°C then
Run Pressure, psig : 1,020 heated to 425°C within 2 hours.

Liquid Hourly Space Velocity : 0.5

H₂: Oil Ratio, scf/bbl : 10,000

Time, Hours: 3 6 9 12

wt% N₂ : .70 .46 .52 .48

wt% S : .22 .35 .25 .33

ASTM Distillation

3 hours Volume of Charge: 50 ml Final Volume: 46.1 ml

Volume: IBP	5	10	15	20	25	30	35	40	44
°F : 191	358	432	475	502	548	579	608	659	702

6 hours Volume of Charge: 50 ml Final Volume: 45.7 ml

Volume: IBP	5	10	15	20	25	30	35	40	45
°F : 201	272	352	423	479	519	562	615	659	682

(Run A-2 continued)

9 hours Volume of Charge: 50 ml Final Volume: 45.1 ml

Volume: IBP	5	10	15	20	25	30	35	40	45
^O F :	196	299	362	421	472	509	554	581	623 650

12 hours Volume of Charge: 51 ml Final Volume: 46.9 ml

Volume: IBP	5	10	15	20	25	30	35	40	45
^O F :	192	330	384	440	478	511	546	584	626 702

Run No. A-3

Catalyst No. NALCO MO-477 1/16"

Metals : 14.0%MoO₃ 3.3%CoO

Surface Area, m²/g : 250

Pore Volume, ml/g : .55

Pore Diameter, (4V/A), Å: 88

Run Temperature, °C : 425₊₅; started at 335°C, then heated to 425°C within 2 hours

Run Pressure, psig : 1,030

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, Hours: 3 6 9 12

wt% N₂ : .60 .56 .70 .64

wt% S : .20 .35 .37 .19

ASTM Distillation

3 hours Volume of Charge: 50 ml Final Volume: 45.6 ml

Volume: IBP	5	10	15	20	25	30	35	40	44
°F : 220	272	338	411	468	501	546	591	639	690

6 hours Volume of Charge: 50 ml Final Volume: 46 ml

Volume: IBP	5	10	15	20	25	30	35	40	45
°F : 193	264	351	416	473	519	559	601	646	710

(Run A-3 continued)

9 hours Volume of Charge: 50 ml Final Volume: 45.4 ml

Volume:	IBP	5	10	15	20	25	30	35	40	44
O _F	:	213	299	381	439	478	510	560	592	642 740

12 hours Volume of Charge: 50 ml Final Volume: 45.9 ml

Volume:	IBP	5	10	15	20	25	30	35	40	45
O _F	:	203	325	389	457	484	530	571	618	666 680

Run No. A-4

Catalyst No. Harshaw HT-400E 1/16" 720A-2-1-1

Metals : 14.8%MoO₃ 2.8%CoO

Surface Area, m²/g : 222

Pore Volume, ml/g : .51

Pore Diameter, (4V/A), Å: 110

Run Temperature, °C : 425₋₅; started at 335°C, then heated to 425°C within 2 hours.

Run Pressure, psig : 1,030

Liquid Hourly Space Velocity : 1 ; catalyst was backflowed with SRC-II feeding at 335 °C.

H₂: Oil Ratio, scf/bbl : 10,000

Time, Hours:	3	6	9	12
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wt% N ₂	:	.90	.72	.94	.88
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wt% S	:	.42	.22	.47	.30
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ASTM Distillation

3 hours Volume of Charge: 51 ml Final Volume: 44.3 ml

Volume: IBP	5	10	15	20	25	30	35	40	43
O _F	: 221	316	405	458	500	551	591	637	671 683

6 hours Volume of Charge: 50 ml Final Volume: 44 ml

Volume: IBP	5	10	15	20	25	30	35	40	42
O _F	: 219	348	406	459	501	548	599	641	679 700

(Run A-4 continued)

9 hours Volume of Charge: 50 ml

Final Volume: 44 ml

Volume: IBP	5	10	15	20	25	30	35	40	42
^O F :	227	346	403	453	498	548	589	643	669 671

12 hours Volume of Charge: 51 ml

Final Volume: 44 ml

Volume: IBP	5	10	15	20	25	30	35	40	42
^O F :	231	329	426	474	514	542	589	634	671 710

Run No. A-5

Catalyst No. MSU-C20-E

Catalyst Composition

Metals : 13.0%MoO₃ .1%NiO .3%CoO

Base : NALCO-78-6008E-1/16"

Base Surface Area, m²/g : 146.95

Base Pore Volume, ml/g : .6841

Base Pore Diameter (4V/A), Å : 186.2

Base Median Pore Diameter, Å : 420.2

Run Temperature, °C : 425₋₅; started at 400°C, then heated to 425°C within one hour.

Run Pressure, psig : 1,040

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.26	.56	.63	.97	.91	1.01	1.03	.63
wt% S :		.28		.28		.325		.322

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 42 ml

Volume	: IBP	5	10	13	20	25
O _F	: 337	398	448	474	511	550

Volume	: 30	35	40
O _F	: 589	619	643

Run No. A-6

Catalyst No. MSU-C8-B

Catalyst Composition

Metals : 9.8%MoO₃ .45%NiO .43%CoO

Base : NALCO-78-6008B-1/16"

Base Surface Area, m²/g : 232.43

Base Pore Volume, ml/g : .7215

Base Pore Diameter (4V/A), Å : 124.2

Base Median Pore Diameter, Å : 137.3

Run Temperature, °C : 425₋₅; started at 350°C, then heated to 425°C within 1.5 hours.

Run Pressure, psig : 1,090

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.57	.80	.62	.75	.60	.71	.72	.64
wt% S :		.25		.25		.18		.17

ASTM Distillation

Volume of Charge: 41.2 ml Final Volume: 36.4 ml

Volume	:	IBP	5	10	15	20	25
O _F	:	290	378	450	482	521	569
Volume	:	30	35				
O _F	:	620	657				

Run No. A-7

Catalyst No. MSU-C5-B

Catalyst Composition

Metals : 18.6%MoO₃ .26%NiO .45%CoO

Base : NALCO-78-6008B-1/16"

Base Surface Area, m²/g : 232.43

Base Pore Volume, ml/g : .7215

Base Pore Diameter (4V/A), Å : 124.2

Base Median Pore Diameter, Å : 137.3

Run Temperature, °C : 425±5; started at 350°C, then heated to 425°C within 1.5 hours.

Run Pressure, psig : 1,060

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.19	-	.12	.75	.67	.70	.63	.51
wt% S :		.29		.25		.29		.20

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 44.3 ml

Volume	:	IBP	5	10	15	20	25
°F	:	329	391	433	465	498	530
Volume	:	30	35	40	42.8		
°F	:	561	597	637	656		

Run No. A-8

Catalyst No. MSU-C4-A

Catalyst Composition

Metals : 9.1%MoO₃ .37%NiO 1.38%CoO 8.2%WO₃

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2

Base Pore Volume, ml/g : .7183

Base Pore Diameter (4V/A), Å : 88.9

Base Median Pore Diameter, Å : 90.2

Run Temperature, °C : 425±5; started at 400°C, then heated to 425°C within one hour.

Run Pressure, psig : 1,070

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.15	.22	.24	.46	.91	.75	.62	.73
wt% S :		.205		.28		.20		.17

ASTM Distillation

Volume of Charge: 49.6 ml Final Volume: 46 ml

Volume	:	IBP	5	10	15	20	25
°F	:	211	320	397	438	477	509
Volume	:	30	35	40	45		
°F	:	541	591	628	643		

Run No. A-9

Catalyst No. MSU-C3-A

Catalyst Composition

Metals : 10.4%MoO₃ 1.16%NiO .11%CoO

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2

Base Pore Volume, ml/g : .7183

Base Pore Diameter (4V/A), Å : 88.9

Base Median Pore Diameter, Å : 90.2

Run Temperature, °C : 425±5

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.04	.31	.37	.51	.58	.625	.59	.70
wt% S :		.22		.32		.22		.29

ASTM Distillation

Volume of Charge: 50 ml

Final Volume: 47 ml

Volume	:	IBP	5	10	15	20	25
°F	:	308	378	415	442	478	507
Volume	:	30	35	40	45	46.7	
°F	:	542	579	515	666	681	

Run No. A-10

Catalyst No. MSU-C2-A

Catalyst Composition

Metals : 12.0%MoO₃ .48%NiO .1%CoO

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2

Base Pore Volume, ml/g : .7183

Base Pore Diameter (4V/A), Å : 88.9

Base Median Pore Diameter, Å : 90.2

Run Temperature, °C : 425_±5

Run Pressure, psig : 1,030

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.04	.18	.30	.33	.18	.45	.53	.67
wt% S :		.20		.28		.24		.264

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 45.3 ml

Volume	:	IBP	5	10	15	20	25
°F	:	263	317	381	437	470	499
Volume	:	30	35	40	45		
°F	:	536	572	609	670		

Run No. A-11

Catalyst No. MSU-C1-A

Catalyst Composition

Metals : 19.4%MoO₃ .18%NiO .43%CoO

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2

Base Pore Volume, ml/g : .7183

Base Pore Diameter (4V/A), Å : 88.9

Base Median Pore Diameter, Å : 90.2

Run Temperature, °C : 430₊₅

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂	: .08	.19	.24	.45	.25	.24	.40	
wt% S	:	.27		.26		.22		

ASTM Distillation

Volume of Charge: 50 ml

Final Volume: 47 ml

Volume	: IBP	5	10	15	20	25
O _F	: 242	326	394	429	460	492
Volume	: 30	35	40	45	46	
O _F	: 530	562	600	651	672	

Run No. A-12

Catalyst No. MSU-C14-D

Catalyst Composition

Metals : 20.7%MoO₃ 1.86%NiO 1.2%CoO

Base : NALCO-78-6008D-1/16"

Base Surface Area, m ² /g	:	211.39
Base Pore Volume, ml/g	:	.7943
Base Pore Diameter (4V/A), Å	:	150.3
Base Median Pore Diameter, Å	:	190

Run Temperature, °C : 425±5

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂	: .29	.405	.41	.45	.43	.555	.52	
wt% S	:	.24	.26	.24	.22	.22	.22	

ASTM Distillation

Volume of Charge: 44.4 ml Final Volume: 40.2 ml

Volume	:	IBP	5	10	15	20	25
°F	:	251	360	421	462	498	540
Volume	:	30	35	38.9			
°F	:	587	627	695			

Run No. A-13

Catalyst No. MSU-C12-C

Catalyst Composition

Metals : 9.5%MoO₃ .44%NiO 1.77%CoO

Base : NALCO-78-6C08C-1/32"

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), $\frac{Q}{A}$: 156.5

Base Median Pore Diameter, $\frac{Q}{A}$: 161

Run Temperature, °C : 435_{±5}

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
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wt% N ₂	:	.39	.405	.83	.96	.83	.83	.84
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wt% S	:		.43		.40		.36	
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ASTM Distillation

Volume of Charge: 12.5 ml Final Volume: 9.2 ml

Volume	:	IBP	5	8.2
O _F	:	320	512	585

Run No. A-14

Catalyst No. MSU-C13-C

Catalyst Composition

Metals : 9.5%MoO₃ .79%NiO 1.6%CoO 9.0%WO₃

Base : NALCO-78-6008C-1/32"

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), $\frac{Q}{A}$: 156.5

Base Median Pore Diameter, $\frac{Q}{A}$: 161

Run Temperature, °C : 430₊₅

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
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wt% N ₂	:	.095	.065	.73	-	.67	.95	-
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wt% S	:		.17	.34		.37	.33	
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ASTM Distillation

Volume of Charge: 20 ml Final Volume: 15 ml

Volume	:	IBP	5	10	15
°F	:	350	452	555	610

Run No. A-15

Catalyst No. MSU-C23-B

Catalyst Composition

Metals : 23%MoO₃ .2%NiO 1.4%CoO

Base : NALCO-78-6008B-1/16"

Base Surface Area, m²/g : 232.43

Base Pore Volume, ml/g : .7215

Base Pore Diameter (4V/A), Å : 124.2

Base Median Pore Diameter, Å : 137.3

Run Temperature, °C : 435₋₅

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
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wt% N ₂	:	.13	.19	.48	.29	.39	.44	.81
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wt% S	:		.195		.21		.22	
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ASTM Distillation

Volume of Charge: 46.2 ml Final Volume: 42.5 ml

Volume	:	IBP	5	10	15	20	25
°F	:	236	322	390	443	478	511

Volume	:	30	35	40	41.5
°F	:	548	595	640	677

Run No. A-16

Catalyst No. MSU-C24-E

Catalyst Composition

Metals : 15.5%MoO₃ .15%NiO 2.3%CoO

Base : NALCO-78-6008E-1/16"

Base Surface Area, m²/g : 146.95

Base Pore Volume, ml/g : .6841

Base Pore Diameter (4V/A), Å : 186.2

Base Median Pore Diameter, Å : 420.2

Run Temperature, °C : 425±5

Run Pressure, psig : 1,040

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
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wt% N ₂	:	.22	.44	.53	.54	.79	.78	.73
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wt% S	:		.19		.22		.26	
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ASTM Distillation

Volume of Charge: 47.2 ml Final Volume: 44 ml

Volume	:	IBP	5	10	15	20	25
°F	:	230	340	411	454	496	528

Volume	:	30	35	40	42.7
°F	:	568	613	659	680

Run No. A-17

Catalyst No. MSU-C9-B

Catalyst Composition

Metals : 8.3%MoO₃ .57%NiO 1.55%CoO 8.8%WO₃

Base : NALCO-78-6008B-1/16"

Base Surface Area, m ² /g	:	232.43
Base Pore Volume, ml/g	:	.7215
Base Pore Diameter (4V/A), Å	:	124.2
Base Median Pore Diameter, Å	:	137.3

Run Temperature, °C : 435±5

Run Pressure, psig : 1,060

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.14	.11	.58	.20	.28	.205	.17	
wt% S :		.17		.19		.19		

ASTM Distillation

Volume of Charge: 25 ml Final Volume: 21.8 ml

Volume	:	IBP	5	10	15	20	20.7
°F	:	181	322	442	516	600	604

Run No. A-18

Catalyst No. MSU-C10-C

Catalyst Composition

Metals : 20.6%MoO₃ .1%NiO 1.08%CoO

Base : NALCO-78-6008C-1/32"

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 435_±5

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
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wt% N ₂	:	.34	.67	.52	.39	.5	.505	.49
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wt% S	:		.26		.17		.24	
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ASTM Distillation

Volume of Charge: 49 ml

Final Volume: 45.2 ml

Volume	:	IBP	5	10	15	20	25
°F	:	183	214	348	416	458	490

Volume	:	30	35	40	45
°F	:	547	575	615	660

Run No. A-19

Catalyst No. MSU-C15-D

Catalyst Composition

Metals : 13.7%MoO₃ .78%NiO .1%CoO

Base : NALCO-78-6008D-1/16"

Base Surface Area, m²/g : 211.39

Base Pore Volume, ml/g : .7943

Base Pore Diameter (4V/A), Å : 150.3

Base Median Pore Diameter, Å : 190

Run Temperature, °C : 435±5

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.35	.45	.33	.53	.48	.595	.69	
wt% S :		.22		.22		.18		

ASTM Distillation

Volume of Charge: 50 ml Final Volume: 46.8 ml

Volume	:	IBP	5	10	15	20	25
O _F	:	253	333	400	440	481	511
Volume	:	30	35	40	45	46	
O _F	:	544	581	620	668	672	

Run No. A-20

Catalyst No. MSU-C16-D

Catalyst Composition

Metals : 11.8%MoO₃ .1%NiO 1.9%CoO

Base : NALCO-78-6008D-1/16"

Base Surface Area, m²/g : 211.39
 Base Pore Volume, ml/g : .7943
 Base Pore Diameter (4V/A), Å : 150.3
 Base Median Pore Diameter, Å : 190

Run Temperature, °C : 440±5

Run Pressure, psig : 1,060

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂ :	.44	.39	.27	.38	.30	.46	.49	
wt% S :		.18		.24		.224		

ASTM Distillation

Volume of Charge: 50.5 ml Final Volume: 45.2 ml

Volume	:	IBP	5	10	15	20	25
°F	:	238	322	389	435	470	506
Volume	:	30	35	40	44.8		
°F	:	548	584	618	680		

Run No. A-21

Catalyst No. MSU-C-27-C

Catalyst Composition

Metals : 11.54%MoO₃ 6.6%CoO 3.16%NiO 9.5%WO₃

Base : NALCO-78-6008C-1/32"

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 425±5

Run Pressure, psig : 1,100

Liquid Hourly Space Velocity : 0.9

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 82.9

Time, min:	30	45	60	75	90	105	120	150
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wt% N ₂	:	.06	.18	.085	.085	.10	.155	.15
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wt% S	:	.16	.22	.18	.20
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ASTM Distillation

Volume of Charge: 47.9 ml Final Volume: 45.9 ml

Volume	:	IBP	5	10	15	20	25
°F	:	171	199	255	364	400	440

Volume	:	30	35	40	45
°F	:	480	550	571	588

Run No. A-22

Catalyst No. MSU-C25-A

Catalyst Composition

Metals : 12.5%MoO₃ 2.66%NiO 6.65%CoO 9.66%WO₃

Base : NALCO-78-6008A-1/32"

Base Surface Area, m²/g : 323.2

Base Pore Volume, ml/g : .7183

Base Pore Diameter (4V/A), $\frac{O}{A}$: 88.9

Base Median Pore Diameter, $\frac{O}{A}$: 90.2

Run Temperature, °C : 425±5

Run Pressure, psig : 1,100; went up to 1,300 psig after 3 hours.

Liquid Hourly Space Velocity : 1 (it was 1.8 LHSV at first 30 minutes)

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 77.9

Time, min: 30 45 60 75 90 105 120 150 180 210 240

wt% N₂ : .14 .18 .11 .14 .13 .15 .14 .15 .22

wt% S : .27 .20 .16 .21 .19 .23

ASTM Distillation

Volume of Charge: 40.3 ml Final Volume: 37.9 ml

Volume	:	IBP	5	10	15	20	25
O _F	:	199	398	415	473	482	529

Volume	:	30	35	37
O _F	:	580	628	661

Run No. A-23

Catalyst No. MSU-C29-E

Catalyst Composition

Metals : 14.4%MoO₃ 3.1%NiO 4.5%CoO 9.1%WO₃

Base : NALCO-78-6008E-1/16"

Base Surface Area, m²/g : 146.95

Base Pore Volume, ml/g : .6841

Base Pore Diameter (4V/A), Å : 186.2

Base Median Pore Diameter, Å : 420.2

Run Temperature, °C : 425+5

Run Pressure, psig : 1,100; went up to 1,250 psig after 4 hours.

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 76.8

Time, min: 30 45 60 75 90 105 120 150 180 210 240

wt% N₂ : .05 .07 .19 .16 .06 .20 .23 .22 .16 .32 .41

wt% S : .18 .35 .20 .17 .19 .40 .19 .24

ASTM Distillation

Volume of Charge: 30.9 ml Final Volume: 29.0 ml

Volume : IBP 5 10 15 20 25
O_F : 192 272 418 473 542 608

Volume : 30 35 40
O_F :

Run No. A-24

Catalyst No. MSU-C26-B

Catalyst Composition

Metals : 13.25%MoO₃ 3.0%NiO 6.7%CoO 9.45%WO₃

Base : NALCO-78-6008B-1/16"

Base Surface Area, m²/g : 232.43
 Base Pore Volume, ml/g : .7215
 Base Pore Diameter (4V/A), Å : 124.2
 Base Median Pore Diameter, Å : 137.3

Run Temperature, °C : 425±5

Run Pressure, psig : 1,050; went up to 1,300 psig after
 3 hours.

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
 : 79.2

Time, min: 30 45 60 75 90 105 120 150 180 210 240

wt% N₂ : .01 0 .06 .05 .12 .10 .30 .32 .14

wt% S : .20 .22 .28 .18 .12 .19

ASTM Distillation

Volume of Charge: 41.6 ml Final Volume: 39 ml

Volume : IBP 5 10 15 20 25
 °F : 188 246 340 406 446 483

Volume : 30 35 38.1
 °F : 538 594 644

Run No. A-25

Catalyst No. MSU-C28

Catalyst Composition

Metals : 12.6%MoO₃ 6.4%CoO 2.87%NiO 7.75%WO₃

Base : NORTON 6176-1/16"

Base Surface Area, m²/g : 250

Base Pore Volume, ml/g : .7

Base Pore Diameter (4V/A), Å : 152

Base Median Pore Diameter, Å :

Run Temperature, °C : 425±5

Run Pressure, psig : 1,050; went up to 1,300 psig after 3.5 hours.

Liquid Hourly Space Velocity : 1

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 77

Time, min: 30 45 60 75 90 105 120 150 180 210 240

wt% N₂ : 0 .02 .10 .09 .19 .14 .32 .24 .24 .35

wt% S : .19 .13 .15 .17 .21 .25 .23

ASTM Distillation

Volume of Charge: 43 ml

Final Volume: 39.0 ml

Volume	:	IBP	5	10	15	20	25
°F	:	170	250	334	400	448	494

Volume	:	30	35	38
°F	:	550	600	648

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