Catalysts for hydrotreating solvent refined lignite
by Nam Kyun Kim

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:
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The liquid products from the reactor were analyzed for nitrogen content. Sulfur content and distillate yield were also analyzed for the selected composite samples.

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The two consecutive thermal regenerations of the best catalyst was completed. The results showed that the catalytic performance can be fully restored.

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ABSTRACT

Catalytic hydrogenation of the solvent refined lignite(SRL) made by the University of North Dakota's Process Development Unit was carried out using thirty MSU-fabricated catalysts. The solid SRL was dissolved in tetralin to use as the feed for the trickle bed continuous reactor. A total of 42 tests were performed including three commercial catalysts and four blank carriers.

The liquid products from the reactor were analyzed for nitrogen content. Sulfur content and distillate yield were also analyzed for the selected composite samples.

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The two consecutive thermal regenerations of the best catalyst was completed. The results showed that the catalytic performance can be fully restored.

INTRODUCTION

National recognition of dwindling petroleum resources and increasing dependency of our energy needs on foreign oil accelerated our effort to find alternative energy sources. Although the United States has more energy available in the form of coal than in the combined sources of petroleum, natural gas, oil shale and tar sands, the primary use of this solid fuel has been limited to the electric power generation where coal was pulverized and burned in solid form in boilers.

Our abundant coal reserve is immediately available for commercial production of synthetic fuel,"synfuel," from coal to meet our national demand for clean energy. President Carter has signed the Synthetic Fuel Corp. legislation, a mandate to produce the equivalent of 500,000 barrels per day (bbl/d) of synfuel by 1987 and 2 million bbl/d by 2000 (1,2). The $20-billion synfuel program, the largest federal energy-assistance effort in history, is designed to encourage the development of oil shale and coal technologies as well as biomass conversion, alcohol fuels production and recovery of heavy oil, tar sands, and gas from unconventional sources.

The coal must be processed into the desirable products that can substitute for those derived from oil and natural gas. These substitute fuels include crude oils, fuel oil, and distillate; chemical feedstocks; pipeline quality and fuel gas; and other solid products such as char and SRL that may be useful in energy production(3).

Numbers of coal conversion processes and technologies have been developed, applied to various domestic coals, and proved to be potential for the commercial production. These conversion processes can be divided into three major categories:

(1) solid clean fuel (SRL, SRC-I, and char)

(2) liquid fuel (direct:SRC-II, EDS, H-coal and etc.; indirect:Fischer-Tropsch, methanol, M-gasoline, and etc.)

(3) gas fuel (CO_2 accepter, Hygas, Synthane, Bigas, Lurgi process, flash pyrolysis, and etc.) (4)

At present there are many processes that can be put into a commercial demonstration based upon the abundant technological data but the manufacturing cost appears to be yet greater than those for conventional petroleum fuel.

The current war in the Gulf and steadily increasing trend of the oil prices forced us to secure commercial mass production of synfuel from coal today. But the technology is available and all of these coal conversion processes could be viable as the cost of imported crude oil continues to rise.

BACKGROUND

Coal is readily combustible rock containing more than 50% by weight and more than 70% by volume of carbonaceous material including inherent moisture, formed from compaction and induration of variously altered plant remains similar to those in peat. The fermentation of vegetable matter under conditions of no air and abundant moisture where volatiles are retained, resulting in formation of bituminous, such as peat and coal, is known as bituminous fermentation.
The peat-to-anthracite theory of coal formation is described as a process in which the progressive ranks of coal are indicative(4).

According to the most recent estimates by the U.S. Geological Survey, the lignite shares a 6% of the demonstrated reserve base (437 billion tons) which is approximately 25% of the identified resources.
The lignite in the Northern Great Plains occurrs in relatively thick seams ranging from 5 ft to more than 100 ft and typically with overburden from 50 to 200 ft (5)(Figure 1).

Significant sulfur and nitrogen reduction is obtained and the mineral substances are removed from

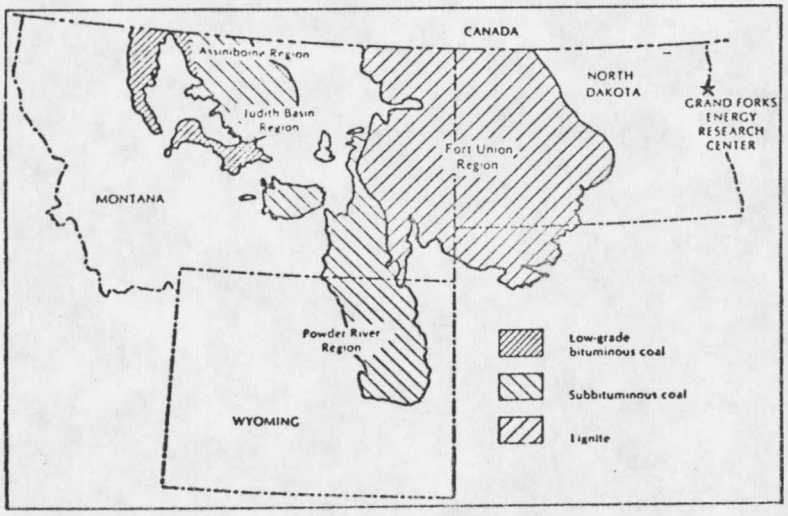


FIGURE 1. NORTHERN GREAT PLAINS COAL PROVINCE

SOURCE: GRAND FORKS ENERGY RESEARCH CENTER, 1978 PROGRAM REPORT

the liquid phase to a low concentration by the SRC-I process.

Project Lignite, equivalent to the SRC-I, was awarded to the University of North Dakota in 1972 for the purpose of determining the appropriate technological approach to the conversion of lignite to clean fuel. The project was expected to extend the two stage conversion of lignite to liquid fuel (equivalent to the SRC-II) with the SRL as an intermediate solid fuel. The first stage to convert lignite to the SRL has been successfully accomplished but the second stage that catalytically hydrotreat the SRL to the distillate fuel was not implemented(6).

Lignite

The classification system adopted by the American Society for Testing and Materials (ASTM) indicates that the lignite is the lowest rank of coals in terms of calorific value and carbonaceous content. These criteria are presented in Figure 2 (7). Lignite coal is dark brown substance that is intermediate in coalification between peat and subbituminous coal. It is consolidated coal with a calorific value less than

8,300 Btu per pound, on a moist, mineral-matter-free basis.

The U.S. Geological Survey established a coal resource base of 3.2 trillion tons, about half of which is considered as recoverable. Of this 1.6 trillion recoverable tons, approximately half (780 billion tons) was determined from actual mapping and exploration to depths of 3,000 ft below ground surface. Approximately 200 billion tons are projected as occurring in bituminous coal and anthracite beds 42 inches or more in thickness and in subbituminous coal and lignite beds of 10 ft or more in thickness. They are generally less than 1000 ft below the surface. Approximately one-half of the 200 billion tons is based on a relative abundance of information(8). A summary of recoverable coal reserves is shown in Table I by rank.

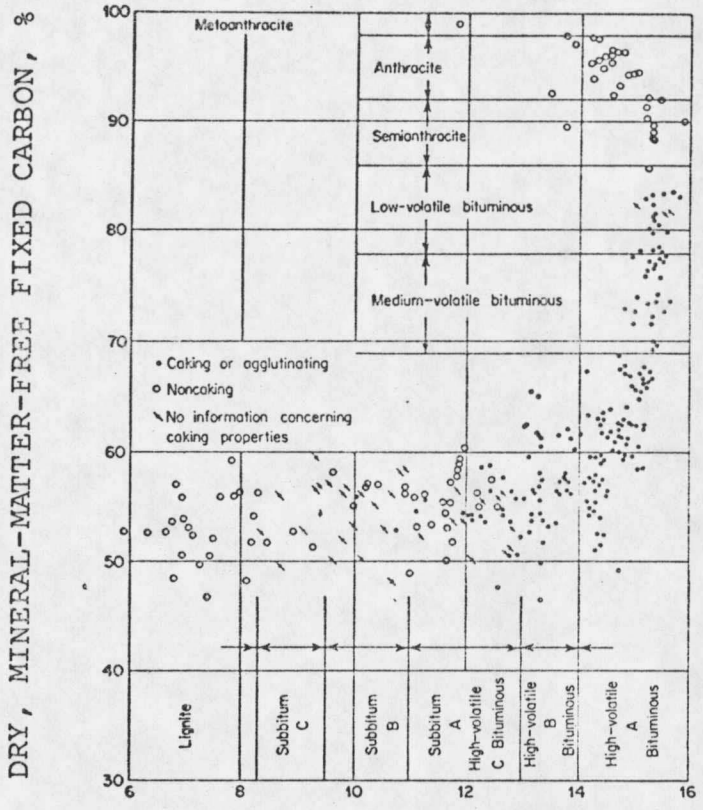


FIGURE 2. DISTRIBUTION PLOT FOR OVER 300 COALS OF THE UNITED STATES, ILLUSTRATING ASTM CLASSIFICATION BY RANK.

Table I

Summary of Recoverable Coal Reserves by Rank
(million tons)

Rank	Strippable	Deep	Total	% of Total Resource
Anthracite	68	2166	2334	10
Bituminous	13,597	54,596	68,340	10
Subbituminous	24,318	32,320	56,227	13
Lignite	8895		8895	2
Total	56,878	89,082	135,696	8

Coal Conversion Processes

Coal conversion processes have been known for nearly two centuries. Gas manufactured from coal was first produced during the late eighteenth century by heating coal in the absence of air. "City gas," a mixture of coke-oven gas, coal, and water gas was a common source of fuel for residential and municipal uses in metropolitan centers for 150 years until replaced by natural gas and electricity (9).

Liquid from coal was first produced in the early 1900s. The most extensive effort in production of coal-derived liquid fuels was that of Germany in World War II. The twelve German plants produced 4.2 million metric tons of liquid fuel from coal during the year 1943-1944 (10).

The methods of coal liquefaction being applied today evolved generally from the major processes: the Bergius process, in which coal was directly hydrogenated to liquid fuels, and the Fischer-Tropsch process, in which hydrogen and carbon monoxide produced from coal were synthesized into liquid fuels. The hydrogenation process was developed more slowly but provided to yield

a much higher grade motor fuel than the gas synthesis process, and by the middle of World War II the German fuel industry was rapidly converting to this type of operation(11,12,13).

Coal conversion plants can be mainly of two different types, gasification and liquefaction. The synthetic gas fuel must have three essential ingredients; carbon from coal, hydrogen from steam, and oxygen from air. Heat for the process may be supplied by combustion of coal. These combustion gases are produced by coal gasification processes; hydrogen, carbon monoxide, and methane. The gas products are determined by the methods of introducing hydrogen, oxygen, and heat into the reactor vessel. Coal gasification process will give one of the three product qualities depending upon the selection of options (Figure 3) (14).

The synthetic liquid fuel from coal requires hydrogen to increase the hydrogen-to-carbon ratio. There are four major approaches to liquefaction. A summary of these approaches is shown in Figure 3(15).

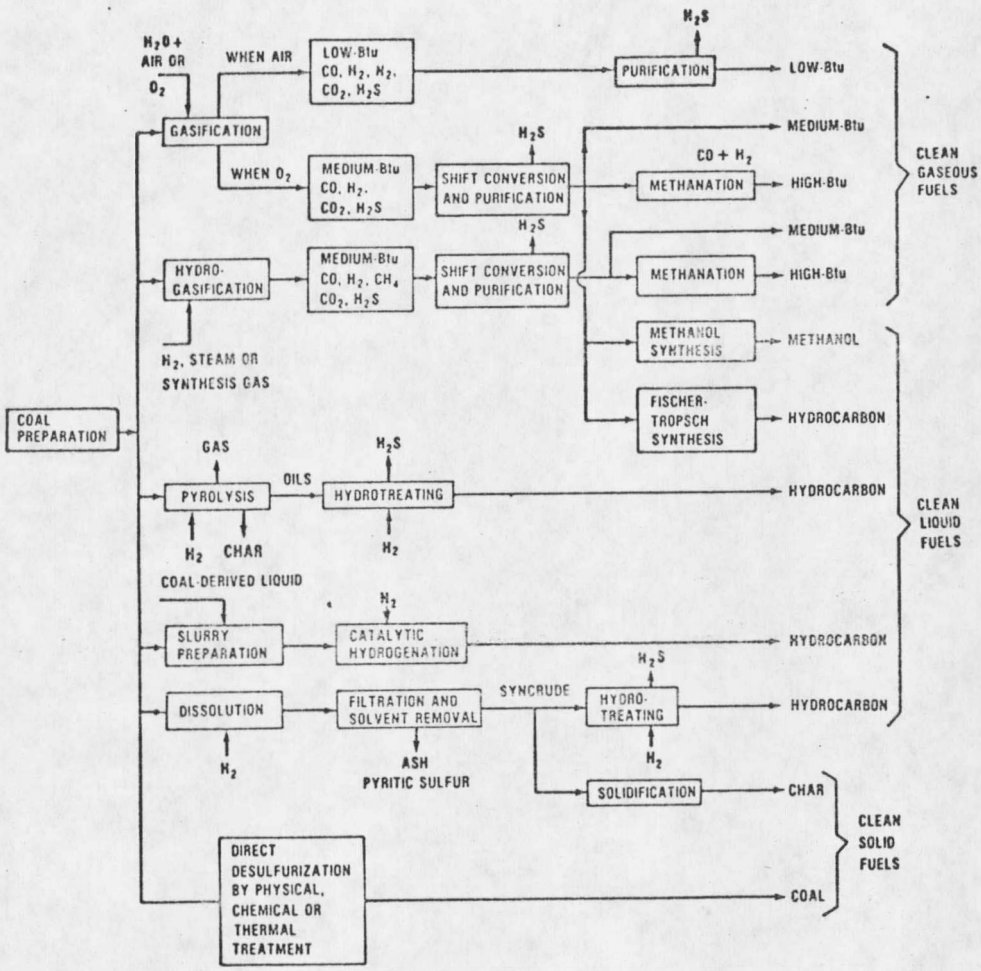


FIGURE 3. CLEAN FUELS FROM COAL

Solvent Refined Lignite

The Project Lignite process development unit (PDU) at the University of North Dakota had a nominal design capacity of 50 pounds of lignite feed per hour. It produced approximately 15 pounds per hour of solvent refined lignite (SRL), solid in the atmosperic conditions(5,6).

Pulverized lignite of full moisture content was slurried with solvent. The Gulf FS-120, a carbon black feedstock, was used as a start-up solvent. The slurry was preheated (around 750^{o}F) and reacted at a selected temperature (normally at 814^{o}F) and pressure (2500 psig) in a reducing gas atmosphere. The products were then separated as gases, liquids, and SRL from the unreacted coal and mineral matters (Figure 4).

The PDU was typically operated on the continuous basis for a 5-day period and data gathered for a number of 12-hour yield periods for material balance purpose. For example, in one run (Run No. M-17AB) 41 pounds per hour (lb/hr) of 31.5% moisture lignite was processed with 536 standard cubic ft per hour (scfh) of gas containing 75% H_2 and 25% CO, 81 lb/hr of process

derived recycle solvent, and 107 lb/hr of toluene recycle in the liquid solid separation system. The yield structure is shown in Figure 5 as a bar graph. On the basis of 100 lb of moisture, mineral-matter-free lignite fed, 65.8 lb of SRL and light organic liquid were produced, and 15.6 lb of CO, 1.0 lb of H_2 and 5.4 lb of water were consumed. Conversion efficiency was over 90% on the moisture, mineral-matter-free lignite basis. The composition and characteristics of the feed lignite and products are shown in Table II(5).

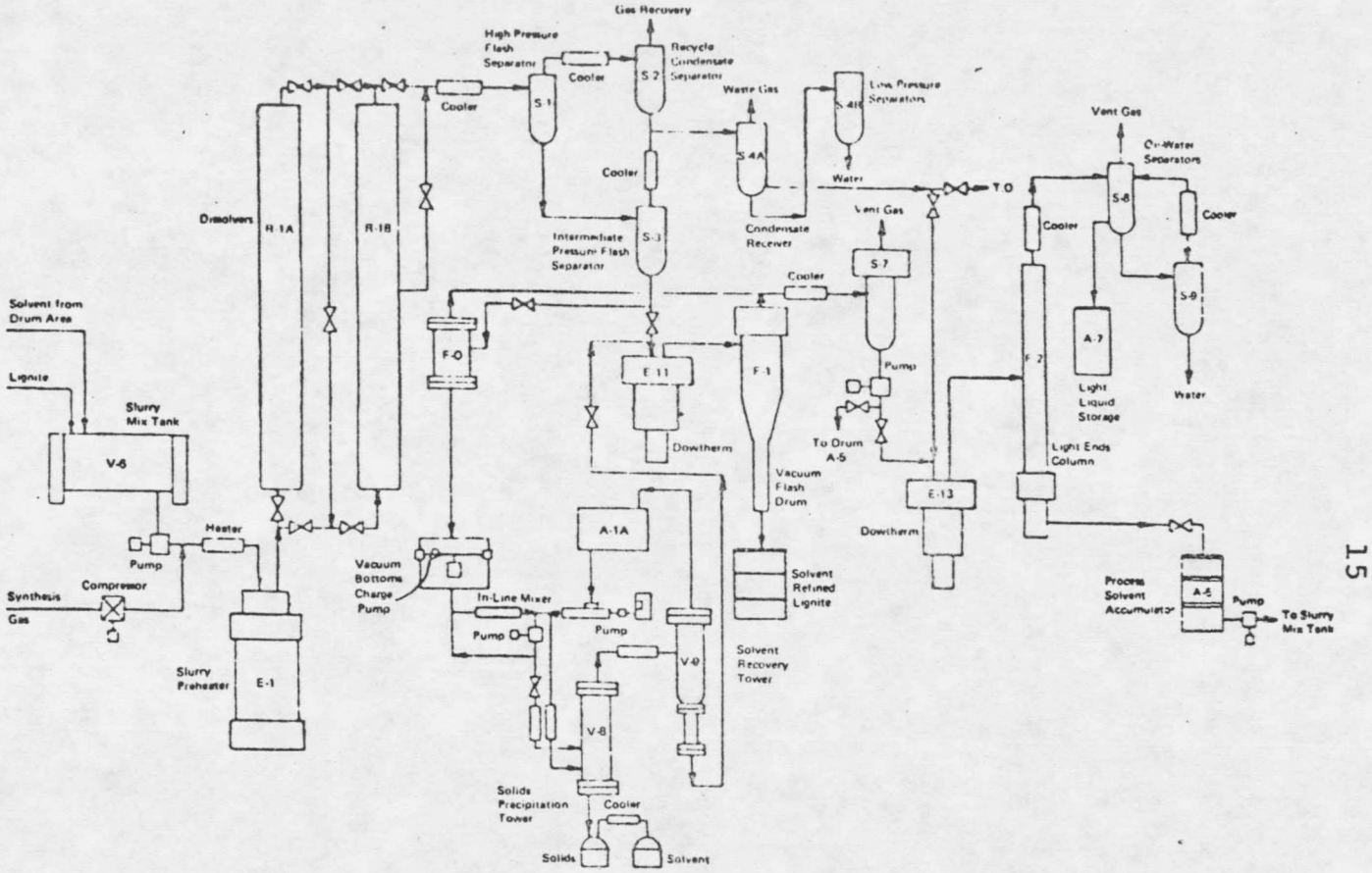


FIGURE 4. SCHEMATIC FLOW DIAGRAM FOR 50 lb/hr PDU, PROJECT LIGNITE.

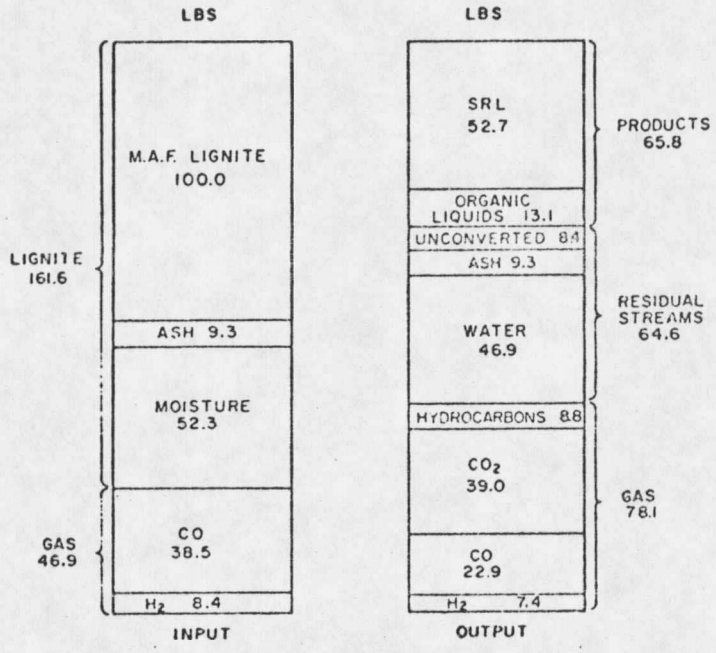


FIGURE 5. BAR GRAPH FOR RUN M-17AB MATERIAL BALANCE

TABLE II

COMPOSITION AND CHARACTERISTICS OF FEED AND PRODUCTS

Feed Gas

	Vol. %	Wt. %
CO	24.7	81.9
H_2	75.2	17.8

Material Balance for Gas Components

	Vol% In	Vol% Out	lb/hr In	lb/hr Out
H_2	75.2	66.2	2.13	1.87
CO	24.7	14.4	9.78	5.81
CO_2	-	13.2	-	9.91
H_2S	-	0.2	-	0.18
CH_4	-	4.5	-	1.11
C_2H_6	-	1.1	-	0.58
C_3H_8	-	0.3	-	0.33
NH_3	-	0.1	-	0.04

Ultimate Analysis of Materials

	Lignite Charged	Starting Solvent	Recycle Solvent	Vacuum Bottoms	Deashed SRL
C	45.22	89.03	83.60	80.20	87.42
H	6.43	8.11	9.14	5.20	5.67
N	0.64	0.12	0.20	0.98	1.07
S	0.45	2.23	1.09	0.90	0.98
O*	41.45	0.51	5.97	4.46	4.86
Ash	5.81	0.0	0.0	8.26	0.0

* By difference

Properties of Product SRL

	Measured*	Calculated
Gradient Bar Melting Point, F	327	-
Pyridine Solubles, wt% ash-free	83.5	100
Specific Gravity	1.28	1.25
Heat of Combustion, Btu/lb	14,330	15,990

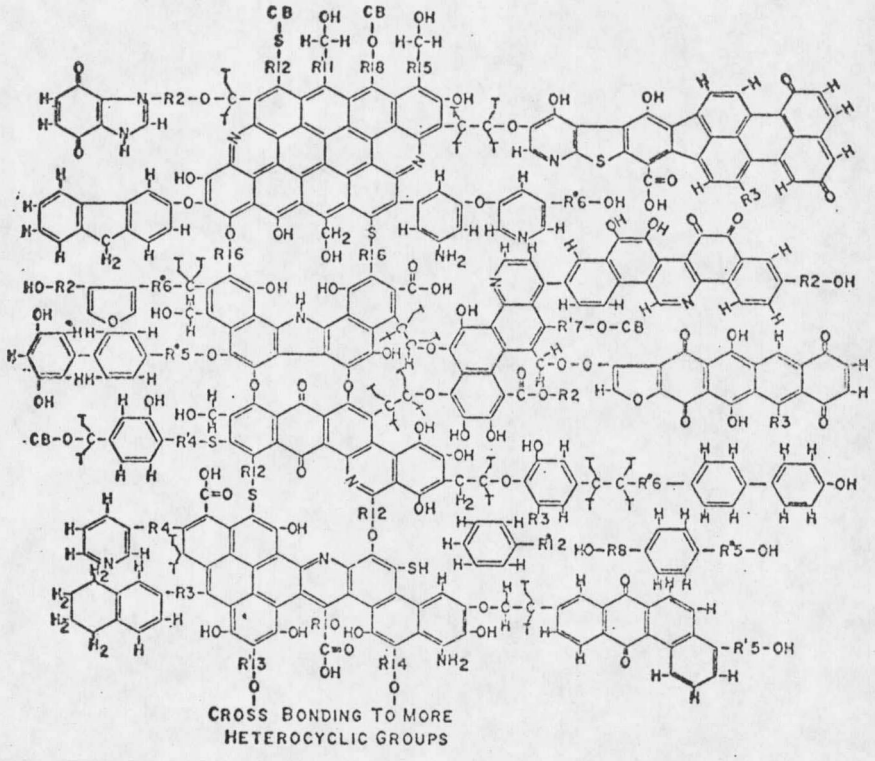
* F-1 vacuum bottoms

Lignite: North American Coal Co., Zap, N.D.
Screen size 90%-200 mesh, 100%-60 mesh
Moisture 31.5%

Chemical Structure of Coal

Coals are comprised of carbon, hydrogen, oxygen, sulfur and nitrogen. Structurally coals are made chiefly of condensed, aromatic rings of high molecular weight. Once coals were thought to be a chicken-wire of 50 to 60 of these ring structures tied together but the nuclear magnetic resonance (NMR) spectra allowed estimates of the distribution of hydrogen atoms between aromatic and non-aromatic structures. About 70% of the carbon atoms are estimated to be in aromatic rings, but only about 23% of hydrogen atoms are attached to aromatic carbon atoms. Hill and Lyon pointed out that it is more logical to assume that only around 10 or less nuclear rings combine for each aggregate(16,17,18). These compounds have molecular weights in the order of 10,000.

An example of the coal molecular structure that can be found in the typical cross-bonded structure of highly volatile coal is shown in Figure 6(16) and a representative of bituminous coal based on the structure given by Hill and Lyon is shown in Figure 7(19). As can be seen from these figures, oxygen, sulfur, and nitrogen



$R^{O}N$=Alicyclic rings of N carbons
RN =Alkyl side chain of N carbon
R'N =Unsaturated alkyl side chain of N carbons
CB=Cross bonding by O or S to new heterocyclic groups with side chains
T=Tetrahedral 3 dimensional C-C bonds and C-S bonds.

FIGURE 6. MOLECULAR STRUCTURE OF HIGHLY VOLATILE COAL (HILL AND LYON)

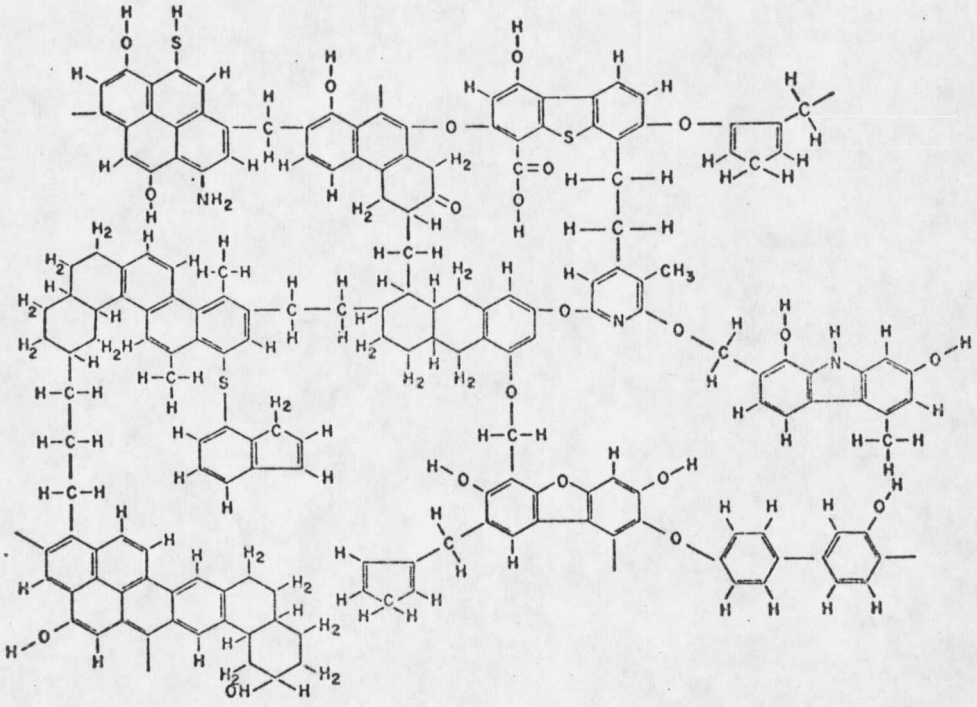


FIGURE 7. A REPRESENTATIVE OF BITUMINOUS COAL

are combined in chemically functional groups, such as OH, CO, COOH, NH_2, CN, S, SH etc., which occurs as an integral part of the original molecules. They also indicate that coal consists of large heterocyclic nuclei monomers with alkyl side chains held together by three dimensional C-C groups, and ether bonds. Sulfur is interchangeable with oxygen in some structures. Nitrogen occurs mainly in the heterocyclic ring structure. The structures tying together the components (aliphatic groups and polynuclear condensed aromatics) constituting coal are considered to be principally aliphatic (methylene) bridges.

Liquefied Coal

Such a complex, heterocyclic structure of coal must be broken down in the reducing atmosphere to obtain desirable products. The major difference between coal and petroleum crudes are shown in Table III(20). A coal with around 0.69 to 0.82 hydrogen atoms per carbon atom must be hydrogenated to produce something richer in hydrogen. The hydrogen/carbon ratio on an atomic basis must be raised to 1.8 to convert coal into syncrude or to 1.94 for distillate type of fuel.

TABLE III

CHEMICAL COMPOSITION OF SOME COALS AND PETROLEUM

	Anthra-cite	Medium volat. bit.	High volat. A bit.	High volat. B bit.	Lignite	Petro-leum crude	Gasol.	Toluene
C	93.7	88.4	84.5	80.3	72.7	83-87	86	91.3
H	2.4	5.0	5.6	5.5	4.2	11-14	14	8.7
O	2.4	4.1	7.0	11.1	21.3	-	-	-
N	0.9	1.7	1.6	1.9	1.2	0.2	-	-
S	0.6	0.8	1.3	1.2	0.6	1.0	-	-
H/C atom ratio	0.31	0.67	0.79	0.82	0.69	1.76	1.94	1.14

Remark: Coal analysis on moisture, mineral-matter-free basis
Ash content of coal 3 to 15%
C-fraction aromatic=0.7. Aromatic rings per cluster-not over 3
H(arom)/H(aliph)=0.23
H/C atom ratio of petroleum residua: asphaltenes 1.18, resin 1.47, oil 1.67

The liquid fuel can be made via liquefaction technologies categorized as direct and indirect. The indirect liquefaction process has a severe disadvantage in terms of thermodynamic efficiency (defined as total output energy divided by total input energy) as well as product efficiency(21). The coal structure does not have to be broken down as is done in the indirect liquefaction process as much of the carbon structure of the coal can be retained in the liquid product at lower energy consumption(22).

The liquefaction process will break many of the bonds as appeared in Figure 6 and Figure 7. Thiophenes, phenols, ethers, methyl pyridines, quinolines, hydroxynaphthalene and pyroles are only a few of the compounds which can be expected to be produced.

Catalytic Hydrotreating for Upgrading

Sulfur, nitrogen, and oxygen contents of coal are often greater than 1% and the oxygen content of lignite is sometimes over 20%. Such heteroatoms are the source of the coal conversion and upgrading problems. Hydrogenation of liquefied coal is slower than that of petroleum crudes because of the abundance of the poly-

nuclear aromatic structure.

Catalytic cleaving of nonhydrocarbon bonds, such as those of nitrogen, oxygen, and sulfur by hydrotreating has been well established in the petroleum refining industries. Some of the commercially proven catalytic hydrocracking method are the Gulf HDS Process, the Union Oil Unicracking Process, and the Standard Oil of California Isocracking Process. Most of these processes prefer the feedstock which contains nitrogen as low as 100 to 400 ppm level(23) but some hydrocracking process (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% (24,25).

Most of the nitrogen atoms present in syncrude is in heterocyclic compounds which are more resistant to hydrodenitrogenation (HDN) than nonheterocyclic nitrogen-containing compounds(aliphatic amines and nitriles) as can be explained by Table IV (26).

Quader et al. indicated that the principal heterocyclic nitrogen containing compounds present in low-temperature coal tars were pyridines, quinolines,

TABLE IV

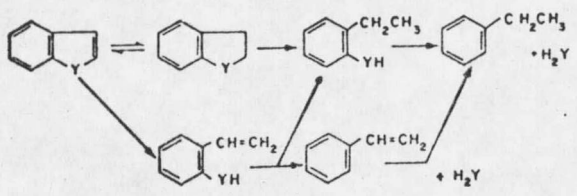
BOND ENERGIES BETWEEN CARBON AND HETEROATOMS IN POLYATOMIC MOLECULES

Bond	Energy (Kcal/mol)
C-H	99
C-C	83
C=C	146
C≡C	200
N-H	93
C-N	73
C=N	147
C≡N	213
C-S	65
C=S	128
S-H	83
C-O	
C=O	

pyrroles, indoles, and carbazoles(27).

Generally, the nitrogen-containing compounds are common in both petroleum and syncrude feedstocks(28), the hydrodenitrogenation (HDN) techniques used for petroleum liquid can be also applied for the coal-derived liquid but due to higher concentrations of nitrogen and aromaticity, it is harder to hydrotreat the syncrude oil (29,30). Hydrodenitrogenation of heterocyclic nitrogen compounds involves saturation of the heterocyclic ring, followed by ring fracture freeing the nitrogen as ammonia. Hydrodesulfurization (HDS) of the heterocyclic ring takes place in the similar fashion to free sulfur as hydrogen sulfide.

Furinsky(31) reported that the relative rates always follow the same trend; that is, the HDS is the fastest, followed by HDN and hydrodeoxygenation (HDO). This can be also explained by the bond energies as shown in Table IV. The basic routes for HDS, HDN, and HDO of such molecules are suggested as shown in Figure 8.



Y = S, O, or NH

FIGURE 8. APPROXIMATE REACTION NETWORK FOR THE REMOVAL OF S, N, AND O FROM FIVE MEMBERED HETERORINGS IN THE PRESENCE OF CATALYST AND HYDROGEN.

Under trickle bed catalytic hydrotreating conditions the denitrogenation is always accompanied by other reactions such as hydrogenation, hydrocracking, desulfurization, deoxygenation, coking, and demetallization. Thermodynamic analysis of the principal steps in reaction of representative heterocyclic nitrogen compounds has been published by Cocchetto et al(32).

Catalysts

Nitrogen content must be reduced to minimize the harmful effect of the subsequent product. Sulfur and oxygen must also be removed to avoid corrosion due to their acidic nature. An appropriate catalyst plays a key role in removing these compounds simultaneously as gaseous ammonia, hydrogen sulfide, and water from the syncrude oil.

Supported molybdate/tungstate catalysts, promoted by either or both cobalt or/and nickel, are most frequently used. The catalytic carrier, quite often alumina, refers to a major catalyst constituent that serves as a base or binder for the active metals and promoters. A carrier may be catalytically active or inert. The major function of a carrier is to give a larger exposed

surface area of active agents for greater catalytic activity than that by themselves in a given amount. It also favorably modifies the catalytic activity or selectivity of the active constituent via catalytic sintering and metal support ineraction(33).

Molybdena and tungstate are both well known active agents for their characteristic activities of HDN, HDS, and HDO(34,35,31).

In order to maintain the catalytic activity, there are two key factors to be considered. The first is the inhibition of coke accumulation by the polymerization of acetylenic residue, the formation of which is thought to require at least three contiguous metal sites. The second is a balance of hydrogenation and hydrogenolysis activity. The latter promotes cracking of surface residues, and the former facile hydrogenation of the cracked species, and therefore inhibits polymerization of carbon species to coke(36).

The ratio of hydrogenation to hydrogenolysis activity and selectivity of a catalyst can be controlled by the appropriate combination of metals and method of catalyst preparation.

Beuther et al. reported that in most commercial appli-

cation the cobalt-molybdenum in atomic ratios varies from 0.1:1.0 to 1.0:1.0 but the best activity was observed for ratios around 0.3:1.0, while a ratio of 0.6:1.0 was best for Ni-Mo catalyst(37).

Ahuja et al. and Kujawa(38,39) concluded that nickel-promoted catalysts are better for HDN than cobalt-promoted catalyst since nickel promotes hydrogenation.

Trickle Bed Reactor

The trickle bed reator is a unit in which a liquid phase and a gas phase flow concurrently downward through a fixed bed of catalyst material to promote desired reaction. Commercial development of the trickle bed reactor can be traced as early as in 1950s to the Shell Company and the British Petroleum Company(40).

Use of trickle bed reactor in the petroleum industry involves the processing with hydrogen of various petroleum materials. The HDS, HDN, and hydrocracking of heavy or residual oil stock to upgrade the quality have been economically tested using this type of reactor(41).

The flow pattern through the fixed bed of catalyst within the reactor is close to plug flow, which is favorable for a high conversion reaction such as HDN at low

liquid-gas ratio. The trickle bed reactor is simple, yet it can handle a high operating pressure and temperature with relatively low capital investment(42).

Operating conditions for HDN and HDS of petroleum feedstocks and Montana State University research runs are shown in Table V. Runnion concluded that the overall best conditions were 445°C, 1000 psig, and a space velocity of 1.0/hr with a H_2/liquid feed ratio of 10,000 scfh(46). Yeh(47) used the same operating conditions except that a temperature of 425°C was used instead. This research adopted operating conditions of 425°C, 1000 psig, 1.0 for space velocity, and 10,000 scf H_2 per barrel of feed.

Table V

OPERATIONG CONDITIONS FOR HYDROTREATING
PETROLEUM AND SYNTHETIC FEEDSTOCKS

	Light Petrol.	Heavy petrol. residu.	COED Liquid	H-coal distill.	Runnion Synthoil
Temp., C	300- 400	340- 420	343- 454	356- 414	400- 450
Press., psig	500-1000	800-2500	2000-4500	800-2500	800-1000
Space Vel., hr^{-1}	2- 10	0.2- 1	0.3- 3	0.44-4.09	1- 5
H_2, scf/bbl	300-2000	2000-10000	-	-	5000-17,000
References	(43)	(43)	(44)	(45)	(46)

Research Objective

The objective of this research is to catalytically upgrade solvent refined lignite (solid SRL) to liquid fuel for immediate industrial use or to clean distillate suitable for conventional refinery feedstock.

Upgrading included reduction of nitrogen and sulfur content of the feedstock and increase in clean product recovered in the ASTM D-86 distillation.

The research plan consisted of preparing the SRL to a manageable liquid phase feed and testing MSU fabricated hydrotreating catalysts in continuous trickle bed reactor. The plan also included regeneration of the best performed catalyst to explore the renewability in the long-run hydrotreating process. Efforts were made to determine why the catalysts performed well as they did.

MATERIALS, EQUIPMENT, AND PROCEDURES

The SRL was dissolved in a solvent to convert into a liquid form. This pretreatment facilitated use of the existing equipment for catalytic hydrotreating. The catalysts were made and tested in the continuous trickle bed reactor to evaluate the performances. The methods of catalyst fabrication and of analyses of the reactor products were described.

Feedstock

The University of North Dakota supplied two gallons of SRL made of Zap lignite and five gallons of SRL made by using Gascoyne lignite. A representative analsis of these two feed stocks are listed in Table VI along with the lignites of two different origins(48,49).

The SRL came in hard-brittle solid of which incipient melting point was observed at approximately 90^{o}C under the Bozeman barometric pressure of 26.5 in. Hg. The first product(Run M-39 of the PDU) of SRL was made using pure hydrogen as compared to 50-50 hydrogen-carbon monoxide syngas used for the second product(Run M-33). Both runs were made at a reactor pressure of 2500 psig and a maximum dissolver temperature of 820-830^{o}F.

Table VI

ANALYSES OF SRL AND NORTH DAKOTA LIGNITES

		Zap Lignite	SRL from F-1 bot. (M-39)	Gascoyne Lignite	SRL from F-1 bot. (M-33)
Quantitative Analysis	Solvent		6.4		2.5
	SRL		92.3		97.32
	Coal	64.70	0.28	54.84	
	Ash	6.56	1.02	8.48	0.18
	Water	28.74		36.68	
	Total	100.00	100.00	100.00	100.00
	Pyridine Solubles		99.72		100.0
Elem. Anal.	Carbon	46.29	87.74	38.28	85.73
	Hydrogen	5.83	6.15	6.83	5.98
	Nitrogen	0.48	0.87	0.51	0.80
	Sulfur	0.29	0.67	0.71	0.94
	Oxygen(by diff.)	40.55	3.55	45.19	6.37
	Ash	6.56	1.02	8.48	0.18
	Total	100.00	100.00	100.00	100.00
Asphal-tene Test	Wt% ash		1.02		0.18
	Wt% unconverted coal		0.28		-
	Wt% preasphaltenes		27.88		21.20
	Wt% asphaltenes		38.34		36.46
	Wt% maltenes & dist. oil		32.48		42.16
	Total		100.00		100.00

The X-ray fluorescence analysis of ash is shown in Table VII. It is worth noting that any of these constituents may play a key role in deactivating of the catalyst(48,49).

Table VII

ANALYSIS OF ASH

Origin	Zap	Gascoyne
Loss on ignition (808^{o}C)	0.4	
SiO_2	20.2	37.6
Al_2O_3	10.5	12.9
Fe_2O_3	10.0	5.6
TiO_2	0.5	0.8
P_2O_5	0.6	0.5
CaO	26.7	20.5
MgO	6.8	6.9
Na_2O	6.7	4.1
K_2O	0.4	0.3
SO_2	17.2	10.8
Total	100.0	100.0

Catalyst Preparation

All MSU-fabricated catalysts tested in this research were made by impregnating active metal components on commercial base, Nalco extrudates. Four metal salts were selected for their solubilities in water needed in the incipient wetness method. They are:

(1) Ammonium molybdate,

$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ of F.W. 1235.9 with 81.4% essay in MoO_3 of M.W. 143.94

(2) Nickelous Nitrate, $Ni(NO_3)_2\cdot 6H_2O$ of F.W. 290.822 with 99.5% purity, M.W. of NiO=74.71

(3) Cobalt Nitrate, $Co(NO_3)_2\cdot 6H_2O$ of F.W. 291.05 M.W. of CoO=94.7326

(4) Ammonium metatungstate, $5(NH_4)_2O.12WO_3\cdot 7H_2O$ of F.W. 3168.67 with 99% purity, M.W. of WO_3=231.8482

The physical property of Nalco support prior to metal impregnation is presented in each data sheet(50). The sequence of metal loading on the Nalco support was consistent throughout the research in the order of Mo, Ni, Co, and W when more than one metal was loaded.

The following steps served as a general guide for catalyst preparation(51):

(1) Evacuating the support

(2) Contacting the support with the impregnating solution

(3) Removing the excess solution

(4) Drying

(5) Calcination and activation

The support was dried in oven at 110°C and calcined at 450°C for 8 hours to be sure that the net support should be weighed correctly. The volume of the impregnating metal solution was determined in such a way that the excess solution could be minimized.

The final metal oxide concentration of a given catalyst by a single impregnation may be calculated from the pore volume and the concentration of solution neglecting selective adsorption(51). For example, the approximate percent MoO_3 is 12

$$\frac{(0.8 \times 0.17)}{(1+0.8 \times 0.17)} \times 100 = 12$$

if pore volume = 0.8 cubic centimeter/gram (cc/gm) and Mo solution concentration = 17%

The wet catalyst was dried by applying a gentle air stream into a rotating jar of 3/4 or 1 gallon size at approximately 7 to 9 revolution per minute (rpm). Following oven-drying for about 8 hours the catalyst was

calcined at 450°C for 8 hours. The calcined catalyst was weighed for final metal composition after cooling to room temperature in a desiccator.

In general the calcined catalyst made for hydrotreating purpose is to be activated in the reducing atmosphere at temperatures as high as those used in the reaction (usually 300 to 500°C)(51). A batch of the catalyst was charged in a 1-in. dia. sulfiding tube with a concentric thermowell for temperature measurement. The tube was heated to 325°C by an electric furnace while a stream of 10% H_2S in hydrogen was passed through the catalyst for at least 12 hours at 2 to 3 cc per second rate (Figure 9).

Nickel and cobalt oxides were reduced to the corresponding sulfides but the non-reducing oxides of the transition metals (molybdenum and tungsten oxides) were activated, resulting in a reduction of the valence of the metal ion(51). The sulfided catalyst was stored in a sealed container.

The exit gas from the sulfiding tube was chemically scrubbed by a series of impingers with a 20% NaOH solution. Extreme caution has been exercised in dealing

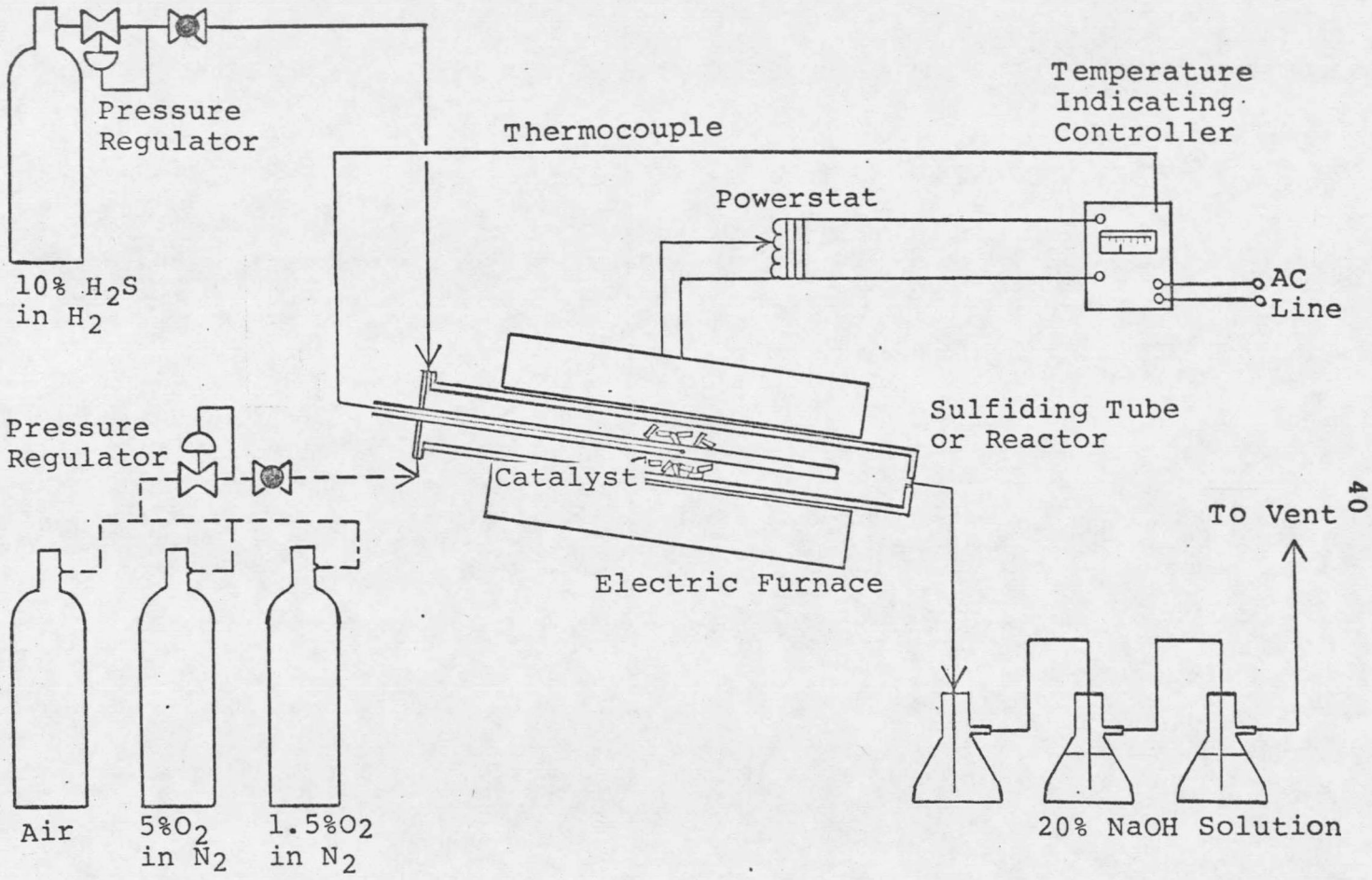


FIGURE 9. ELECTRIC FURNACE AND AUXILIARY EQUIPMENT FOR SULFIDING AND REGENERATION OF CATALYSTS

with hydrogen sulfide. The low concentration (20 to 150 ppm) can be identified by an odor similar to rotten egg. Exposures of 800 to 1000 ppm may be fatal in 30 minutes, and higher concentrations are instantly fatal. H_2S does not combine with the hemoglobin of the blood; its asphyxiant action is due to paralysis of the respiratory center(52,53).

Continuous Trickle Bed Reactor

The bench scale trickle bed reactor used for this research was previously fabricated by the Chemical Engineering Department at Montana State University. A schematic diagram of the trickle bed reactor and auxiliary equipment is shown in Figure 10.

The reactor consisted of a 1-in. I.D. schedule-80 Inconel pipe 35 in. long. The top of the reactor was welded with a ¼-in. stainless steel cross which allowed the fitting of a 33-in. piece of stainless steel tubing, which served as a thermowell, and the fitting of two feed ports, one for the SRL feed and one for the hydrogen. The reactor was fitted into the bore of a 6-in. O.D. aluminum block which was about 3 ft long. The aluminum block was wrapped with three sets of NiChrome wire heating coils encased in ceramic beads. Each heating coil was connected to a Powerstat variable autotransformer which allowed manual temperature control. An iron-constantan thermocouple wire was placed in the ¼-in. stainless steel thermowell at the center of the catalyst section. The thermocouple wire was connected to a Leeds and Northrup continuous stripchart recorder.

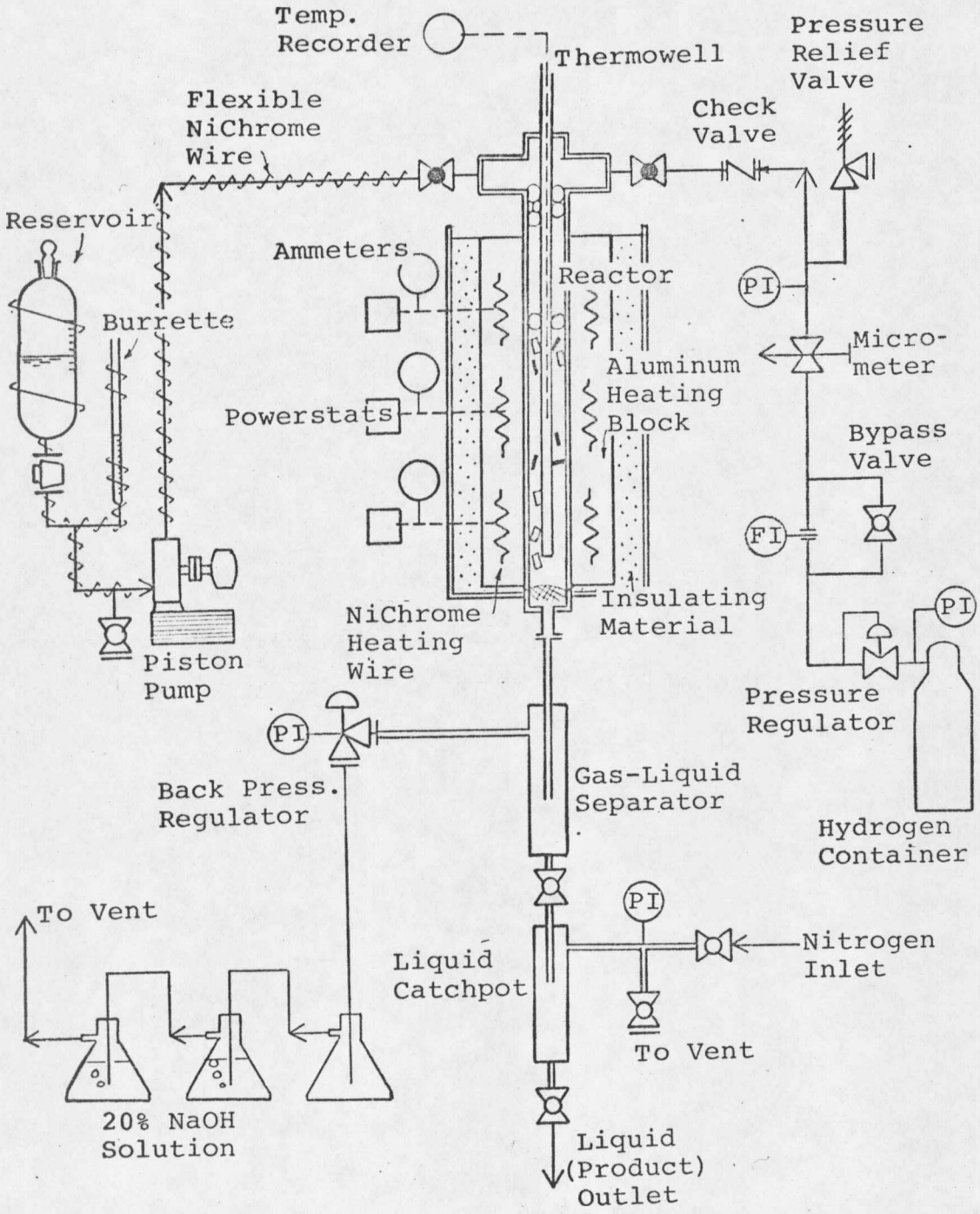


FIGURE 10. TRICKLE BED REACTOR AND AUXILIARY EQUIPMENT

For loading the empty reactor was secured upside-down by a vise. The top of the reactor was loaded with 160 cc of ¼-in. Norton denstone(54) and followed by 25 cc of 1/8-in. denstone (inert ceramic pellets) to serve as the preheating and even-distributing section. Sixty cc of catalyst diluted with 60 cc of 1/8-in. denstone were loaded as the catalyst section(55). Additional 50 cc of 1/8-in. denstone filled the bottom section of the reactor. Finally a stainless steel screen, acted as a plug-support, was placed in the bottom of the reactor before the ¼-in. I.D. reactor closure was threaded into the pipe.

The preheated SRL feed was metered into the top of the reactor by use of a Milton Roy Model A (MR-1-23-49SM) simplex piston pump through a 1/8-in. stainless steel tube. The pump speed was manually controlled by a micrometer. All feed lines and the reservoir were wrapped with flexible heating cords(56) so that the feed temperature was around 130^{o}C by use of Powerstats.

Technical grade hydrogen stored in cylinder was metered through a pressure regulator, a Brooks thermal mass flowmeter, and a micrometer(57).

Gases and liquids were passed through the pressurized reactor (1000 psig) by gravity to a gas-liquid separator. The gases passed through a condenser (air-cooled) and through a Grove back-pressure regulator equipped with a corrosion-resistive Teflon diaphragm. The exit gases were scrubbed by a 20% NaOH solution prior to being vented. The liquids passed from the gas-liquid separator into a catchpot. When a liquid sample(product) was desired, the valve between the separator and catchpot was closed to release the pressure from the catchpot. The catchpot was repressurized with nitrogen and the valve between the separator and the catchpot was reopened connecting the isolated catchpot back on stream.

Operation of Continuous Trickle Bed Reactor

The reactor loaded with the catalyst to be tested was placed in the aluminum heating block. The liquid feed and hydrogen lines were connected at the top of the reactor and the catchpot system was attached at the bottom. The thermocouple wire was inserted into the thermowell. All pipe fittings were sealed using Teflon tape and/or Silver Goop paste, when necessary. The entire system excluding the pump was pressurized to

a normal operating pressure of 1000 psig and checked for leaks using Snoop soap solution. If no leak was detected, the system was depressurized. Three Powerstats were turned on to heat reactor to 425°C.

When the reactor reached desired reaction temperature, the SRL feed, all feed lines, reservoir with a burrett, check valves, and piston housing of the pump were preheated with flexible NiChrome wires controlled by Powerstats. Hot SRL feed was recirculated for a few minutes through the SRL feed line back into the reservoir prior to connecting to the top of the reactor for run. The reactor was pressurized with hydrogen by wide-opening of the bypass valve attached to the Brooks flowmeter. When the system approached desired pressure, the bypass valve was closed and the micrometer was adjusted to keep the hydrogen flow at a rate of 10,000 scf/bbl.

The liquid feed valve located at the top of the reactor was then reopened. The pump started and the time was logged. The feed rates were measured by timing the liquid level drop in the burrett. The liquid feed rate was adjusted by use of pump micrometer to a liquid hourly space velocity(LHSV) of 1 and frequently checked to

ensure a steady flow. The first product was taken in 30 minutes after the start-up and the remainder of samples were collected at 15 min. intervals, unless otherwise specified.

After the last sample was obtained, the pump was shut off and the liquid feed valve at the top of the reactor was closed. The liquid feed line was disconnected and inserted to the top of the reservoir for cleaning solvent recirculation. The excess of the SRL feed was drained. The hydrogen supply was cut off and the hydrogen line was disconnected. The Powerstats were shut off and the catchpot system was removed following reactor depressurization. The reactor was removed from the aluminum heating block, while hot, and the entire content of the reactor was drained in an orderly manner on the plywood for further inspection.

The reactor was brushed using acetone to remove any deposit. Another reactor (No. 0) was especially reserved for Run 30, in which one of the best MSU-fabricated catalyst was tested to evaluate its long-run performance. In this case, the toluene was used to rinse much of the excessive feed from the reactor and

reservoir.

Analytical Procedures

Liquid products from all runs were analyzed for nitrogen and selected odd numbered samples were analyzed for sulfur. If possible, the extent of hydrocracking was measured.

The macro-Kjeldahl method was used to determine nitrogen content using 0.5 gm of product sample and 40 gm of potassium sulfate(58,59,60).

Sulfur concentration was determined by the quartz tube combustion method using a Bico Brown Shell design sulfur apparatus(61,62).

Weight percent denitrogenation(%DN) and weight percent desulfurization(%DS) were calculated as follows;

$$\frac{Fns - Pns}{Fns} \times 100 = \%DN \text{ or } \%DS$$

where Fns = nitrogen or sulfur wt% in the feed

Pns = nitrogen or sulfur wt% in the products

The extent of hydrocracking was determined by ASTM D-86 atmospheric distillation. Fifty cc of composite sample representing for each run were used as a standard test volume, if possible. This method measures the cumulative amount of distilled product which boils below

700°F (or 370°C) or when decomposition begins, whichever occurs first(63).

RESULTS AND DISCUSSIONS

A total of forty two runs have been carried out with the trickle bed continuous reactor using the solvent refined lignite(SRL) dissolved in an organic solvent, 1,2,3,4-tetrahydronaphthalene(tetralin), as the feed.

Thirty MSU-fabricated catalysts were tested to evaluate their performances particularly in denitrogenation along with four blank supports and three commercial catalysts. The remainder of the runs were made for regeneration of the best catalyst.

Preparation of Feed Solution

Chunks of SRL were heated beyond the melting point (approximately 90°C) to 200°C using an electric hot plate but the existing reactor feed system was not equipped to maintain this temperature. Attempts to directly feed the molten SRL to the reactor were unsuccessful due to solidification in the feed line.

The solid SRL was then crushed in a mortar and dissolved in tetralin, while heating and agitating with a magnetic stirrer and heater. The initial ratio of SRL to tetralin was 1:1 for the first and second run. Problems were encountered with pumping of the resulting

feed solution into the reactor due to the insolubles. In an effort to improve the dissolution the ratio of SRL to tetralin was reduced from 1:1 to 1:1.5 for the third and fourth run. However, the additional solvent did not alleviate the pumping difficulties.

Efforts were then made to filter the raw solution through a 200-mesh screen but the excessive insoluble particles blinded the screen.

Finally, the raw solution was centrifuged at 2200 rpm for about 30 seconds. The supernatant which comprised approximately 62% by volume was used as feed for the fourth and fifth run. This almost eliminated the pumping problem.

The SRL to tetralin ratio was further reduced to 1:2 for the use of the fifth run. At this time, the heating period was extended to 72 hours, as compared to a normal one hour. However, the extended heating and additional solvent could not eliminate the need of centrifuge treatment. Therefore, the raw feed solution was centrifuged for the remainder of the runs.

The bottom portion of the centrifuged solution was washed with acetone and dried for ash analysis.

The loss on ignition at 700°C averaged 92% by weight. The remainder of 8% appeared to be ash.

Calibration of Temperature

The temperature recorder with a type "J" thermocouple was calibrated using two typical substances; freezing and boiling point of water and boiling point of glycerol. In addition to these a pure zinc metal was introduced having a melting point of 419°C which is close to the desired reactor operating temperature of 425°C. Later, a Digi-Sense thermometer (Cole-Palmer Model 8520-50) was added for a quick calibration purpose.

Testing of Catalysts

The data sheet for individual run is attached in the appendices in a chronological order. Each data sheet includes a summary of operating conditions, analytical results, characteristics of catalyst and support, and arrangement of the reactor content. The results of Runs 1 through 18, Run 23, Run 41 and Run 42 are shown in Table VIII.

The first run was made using the MSU C30D catalyst. The percent denitrogenation and desulfurization plotted against time on stream are shown in Figure 11.

Table VIII

COMPOSITION AND PERFORMANCE OF VARIOUS CATALYSTS

Run No.	Catalyst No.	(Base)	MoO_3	NiO	CoO	WO_3	% DN	% DS	Yield
1	MSU C30D	Nalco D	10.7	7.16	3.06	8.48	100.0	44.4	57.0
2-5	MSU C33B	Nalco B	12.4	2.70	7.07	10.08	81.6	20.6	47.0
6	MSU KN D1	Nalco D	6.54	2.22	7.06	9.48	48.6	52.7	71.0
7	MSU KN A1	Nalco A	8.42	1.29	4.00	10.04	41.0	70.9	76.4
8	MSU KN B1	Nalco B	8.91	2.28	4.28	9.93	61.1	71.3	71.3
9	MSU KN E1	Nalco E	6.09	2.26	4.00	10.32	58.3	60.5	89.2
10	MSU KN A2	Nalco A	11.0	2.70	4.84	10.20	77.3		68.7
11-12	Blank	Nalco C	0.	0.	0.	0.	61.6		59.2
13	MSU KN D2	Nalco D	11.48	2.84	5.10	10.19	44.4		57.1
14	Blank	Nalco E	0.	0.	0.	0.	58.6		64.2
15	Blank	Nalco C	0.	0.	0.	0.	80.0		57.0
16	Blank (¼")	Denstone	0.	0.	0.	0.	31.8		80.8
17	MSU KN B2	Nalco B	11.1	2.62	4.86	10.47	75.3	19.0	67.5
18	MSU KN E2	Nalco E	10.18	2.84	4.88	10.68	68.2	38.8	70.4
23	Mobil TCC	Dura Bead	0.	0.	0.	0.	83.9		54.2
41	Cyanamid	HDS-20A	16.2	0.	5.0	0.	93.9	86.3	58.2
42	Shell 324		13.0	3.0	0.	0.	95.9	89.2	61.8

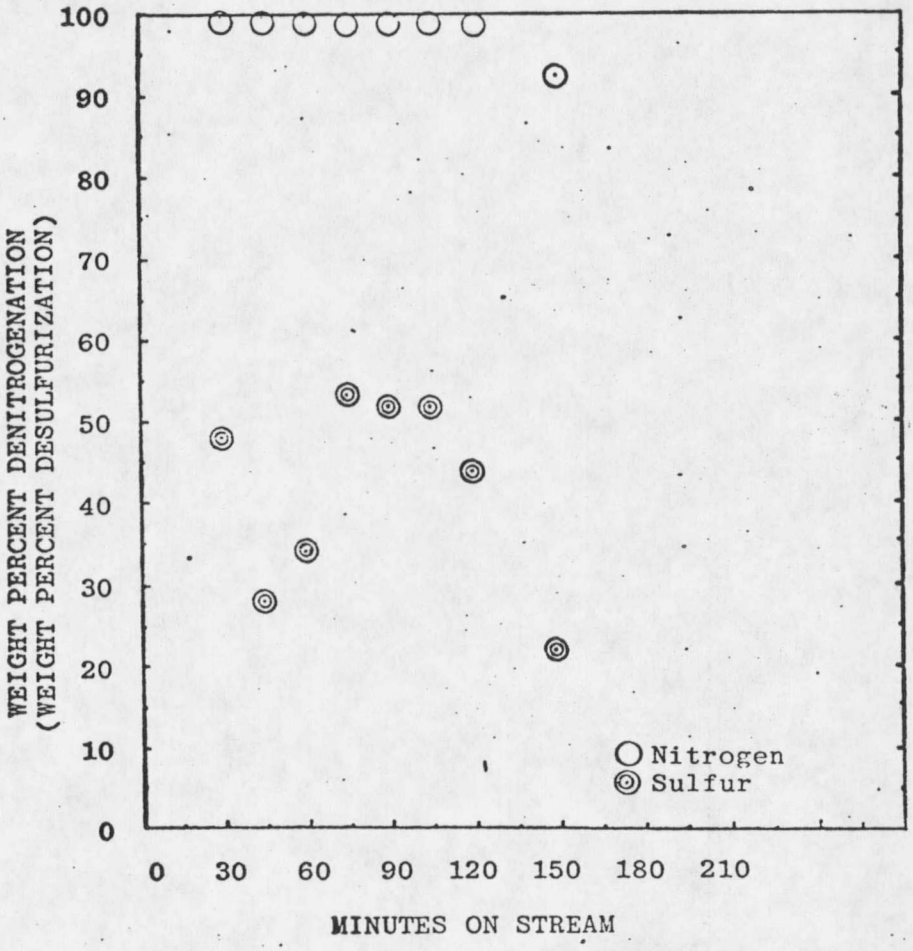


FIGURE 11. DENITROGEN AND SULFUR REMOVAL USING MSU C30D CATALYST (RUN 1)

The nitrogen was virtually eliminated for the first two hours, although the sulfur removal was not as good as the nitrogen removal. The first run was terminated in two and a half hours due to a large fluctuation of pressure (1050 psig to 2100 psig). Unfortunately, the catalyst was discarded without attempting to regenerate. It was believed that the high pressure had been caused by the inconsistent feed rate attributable to the lack of experience.

The MSU C33B catalyst was used next for the second through fifth run; the second and third run were made with the new catalyst, the fourth run with the same catalyst which had been thermally regenerated for the first time and the fifth run with the second-regenerated catalyst. Run 2 lasted only an hour when the reactor pressure increased to 1300 psig. Following a flush with toluene, Run 3 was made for almost 7 hours, completing the last part of catalytic performance. The percent denitrogenation of the products obtained from Run 2 through Run 5 was plotted against time on stream in Figure 12.

In contrast to the first catalyst the percent denitrogenation deteriorated at a faster rate to 50%

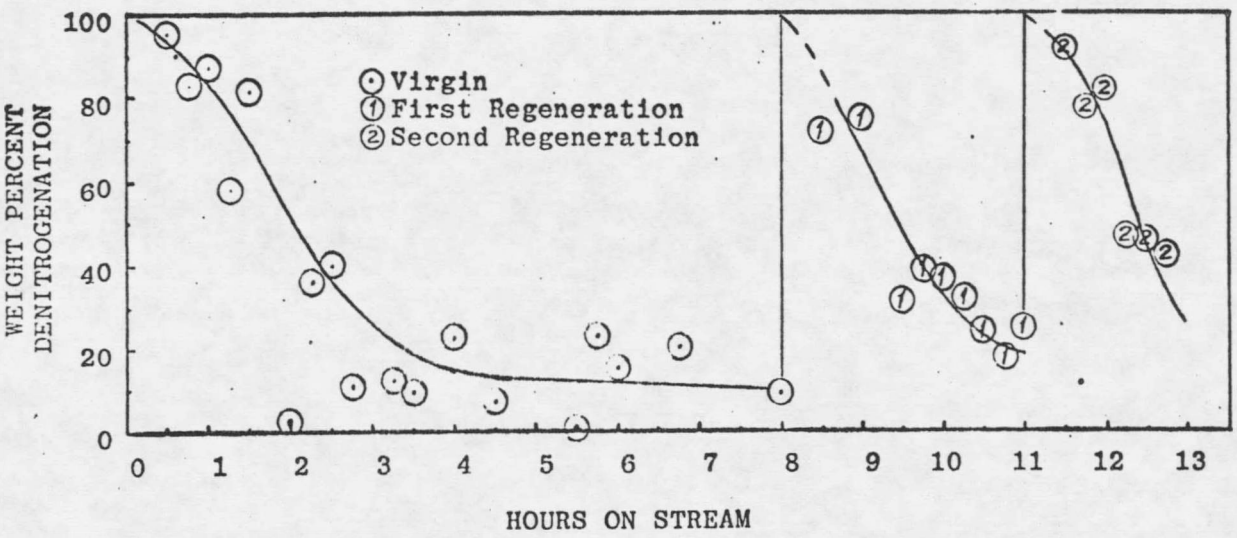


FIGURE 12. NITROGEN REMOVAL USING MSU C33B CATALYST FROM THE VIRGIN TO THE SECOND SUCCESSIVE REGENERATION.

level in two hours. However, the two consecutive thermal regeneration demonstrated that that the denitrogenation and desulfurization capacities could be restored. It must be pointed out that the thermal regeneration procedure was not refined at this time and few incidents of sharp temperature increases up to 900°C were experienced with air.

In view of the results of Run 2 through Run 5 the catalyst was fairly active within the first two hours of reaction, a period of two hours was in general set as a cutoff point. The results of Run 6 through Run 10, Run 13, Run 17, and Run 18 were obtained with eight newly fabricated catalysts. These runs were made to evaluate the effect of different Nalco bases on the catalytic performance.

Run 11, Run 12, and Run 15 were made with Nalco blank bases to see if the bases alone could contribute to the catalytic performance. Figure 13 and Figure 14 showed that the bases had some capacity to remove the nitrogen from the feed solution. The average denitrogenation obtained with the MSU KN E1 catalyst for the first two hours of Run 9 was much the same as that of Run 14

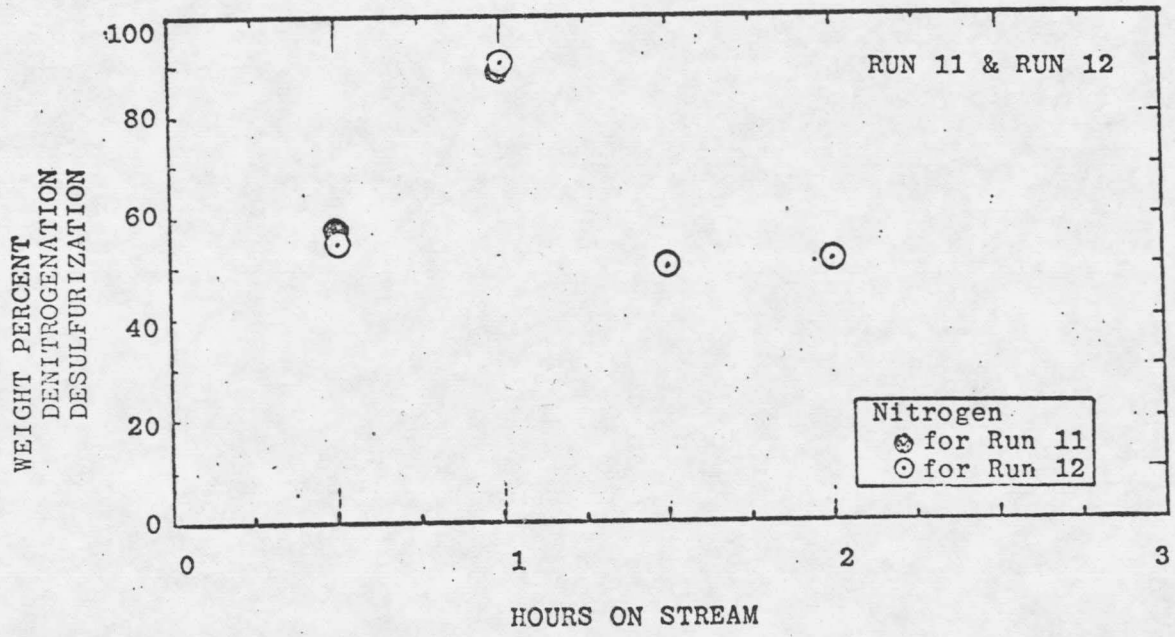


FIGURE 13. EFFECT OF BLANK BASE (NALCO C) IN DENITROGENATION

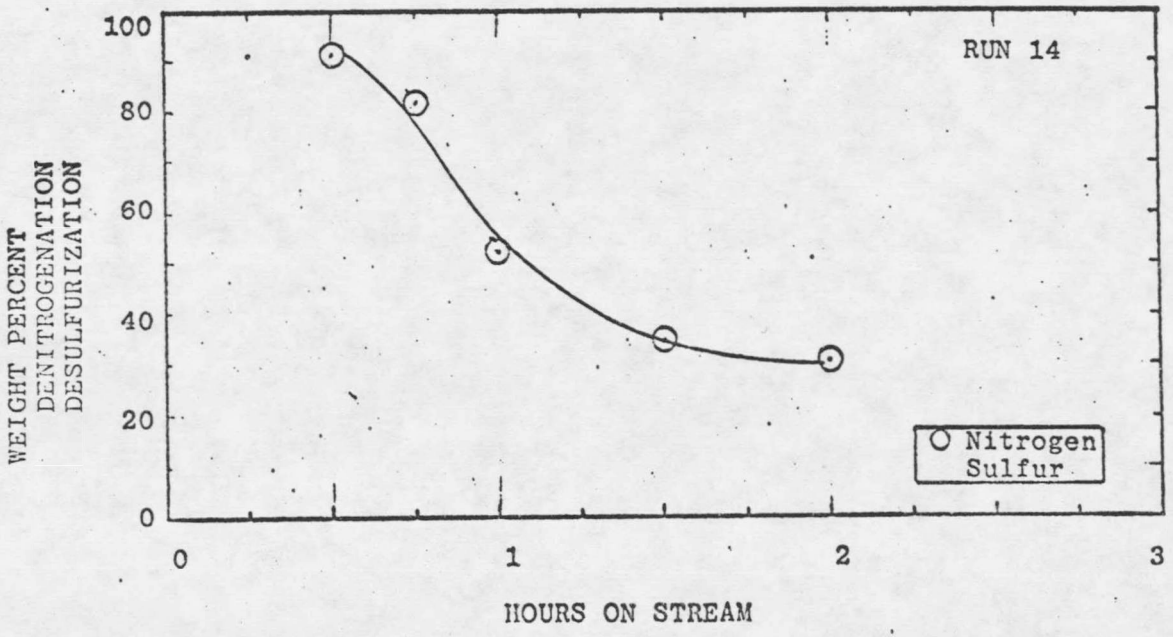


FIGURE 14. EFFECT OF BLANK BASE (NALCO E) IN DENITROGENATION

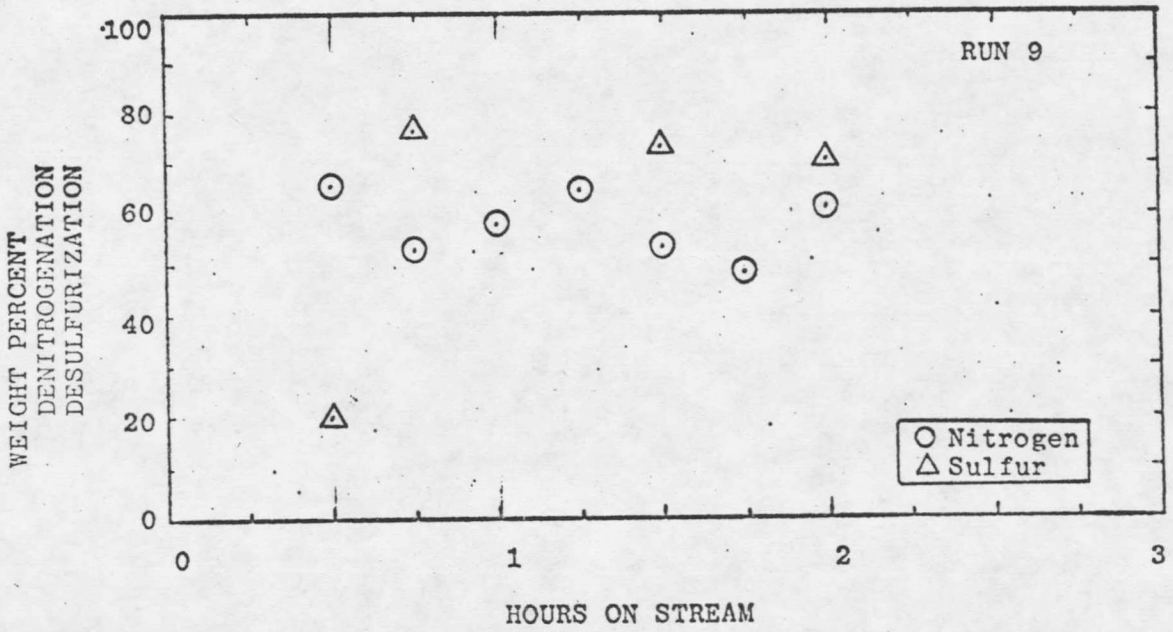


FIGURE 15. PERFORMANCE OF MSU KN E1 CATALYST IN DENITROGENATION AND DESULFURIZATION.

obtained with Nalco base E, although the nature of the percent denitrogenation curve were quite different from each other. The catalyst showed a consistent performance, while the blank base deteriorated fast as shown in Figure 14 and Figure 15. Finally, the entire reactor was filled with ¼-inch Denstone for Run 16. The results of the nitrogen analysis showed the poorest nitrogen removal of all runs.

Occasionally the first few samples of the runs such as Run 10, Run 11, and Run 12, showed poor removal of nitrogen or sulfur. This is probably due to incomplete cleaning of the reactor and to the trace of hydrogen sulfide trapped in the pores of the catalyst during the sulfiding process. This was reduced by thorough cleaning and by purging the reactor with hydrogen for 10 minutes prior to each run.

Run 10, Run 13, Run 17, Run 18, and Run 27 were selected to evaluate the effect of base on nitrogen removal, assuming that the composition of metal oxides was about the same. Figure 16 showed that the percent denitrogenation was affected by the pore volume. The point D outlied from the curve was probably due to a

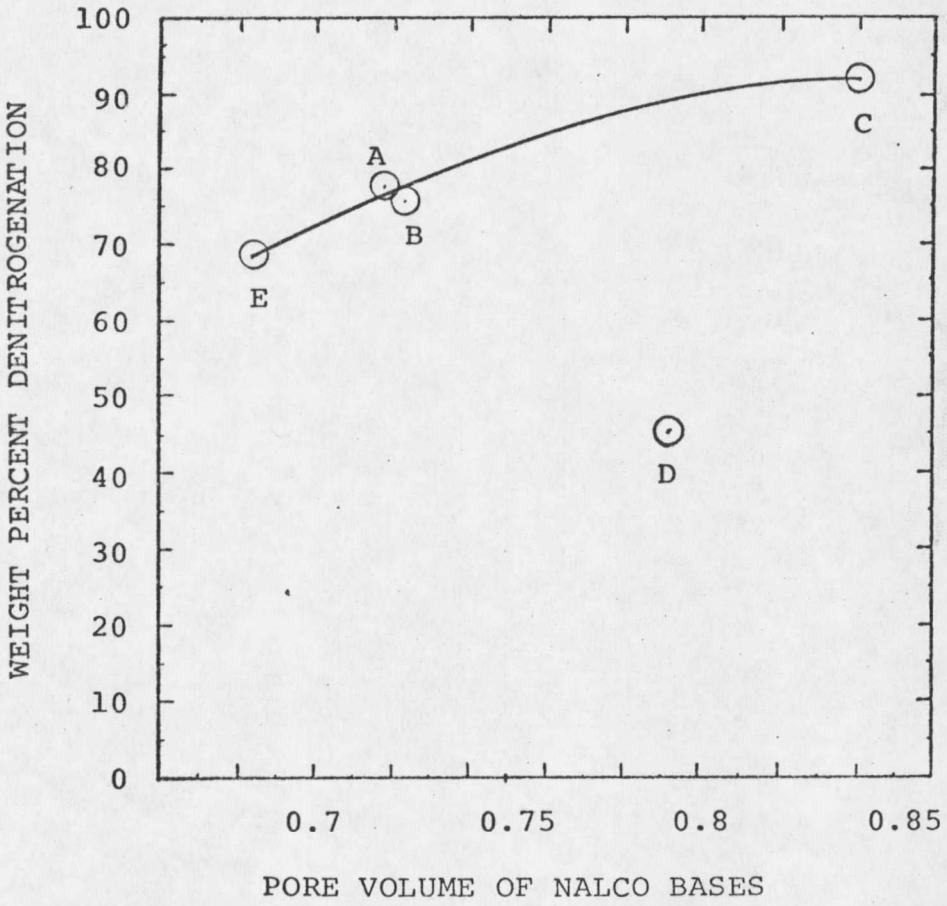


FIGURE 16. EFFECT OF PORE VOLUME ON THE DENITROGENATION

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

different way of catalyst loading in the reactor as shown in the Run 13 data sheet. The catalyst for Run 13 was loaded all in one section instead of diluting with 1/8-inch Denstone. The percent denitrogenation was shown as a function of surface area of the catalyst (Figure 17), as a function of median pore diameter (Figure 18), and as a function of average pore diameter (Figue 19). Figure 20 showed a correlation between the product yield and pore volume.

In general it appeared that the percent denitrogenation improved as the pore volume increased, while the surface area of the catalyst having approximately 200 m^2 per gm was the best.

Run 15 differred from Run 11 in that the LHSV was reduced from 1 to 0.18 by filling 330 cc of Nalco base C instead of a normal volume of 60 cc.

The MSU KN B2 catalyst used for Run 17 averaged 75% denitrogenation as compared to 61% obtained with the MSU KN B1 catalyst. Better nitrogen removal was contributed by an increased amount of MoO_3 (11% vs. 9%). The MSU KN E2 catalyst with 10% MoO_3 also displayed a slight improvement in nitrogen removal over the MSU KN E1

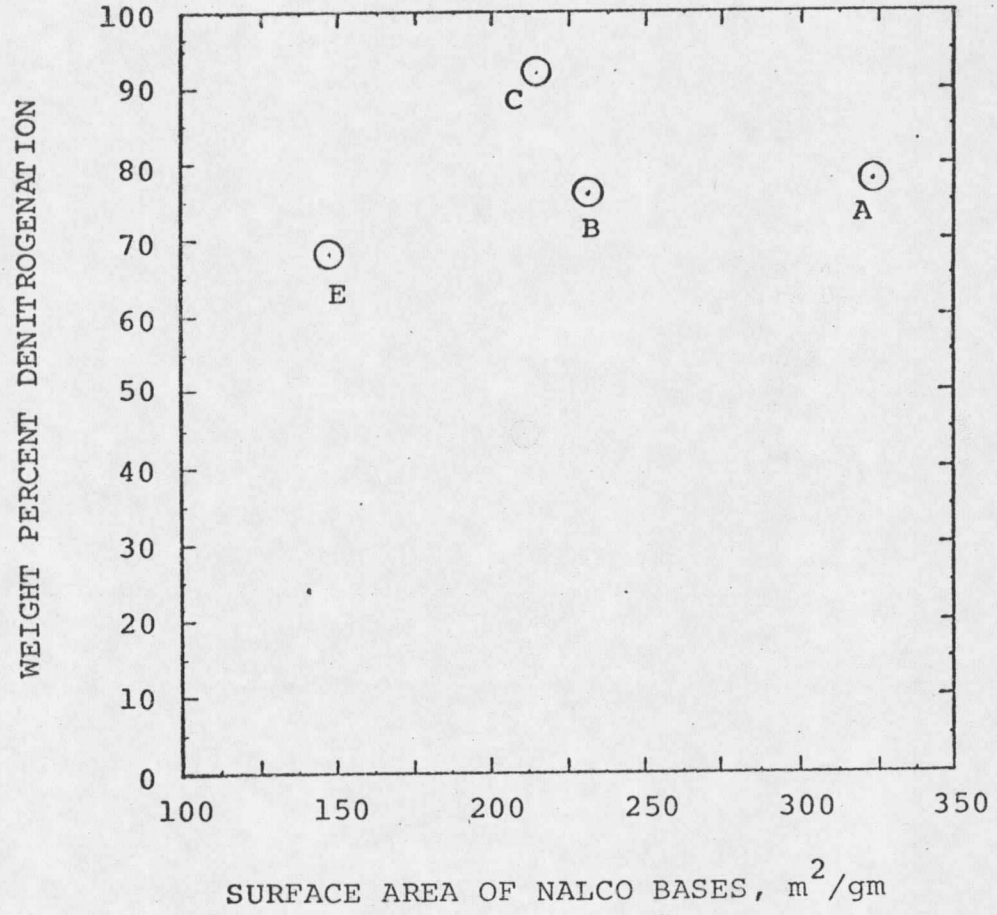


FIGURE 17. EFFECT OF SURFACE AREA ON THE DENITROGENATION

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

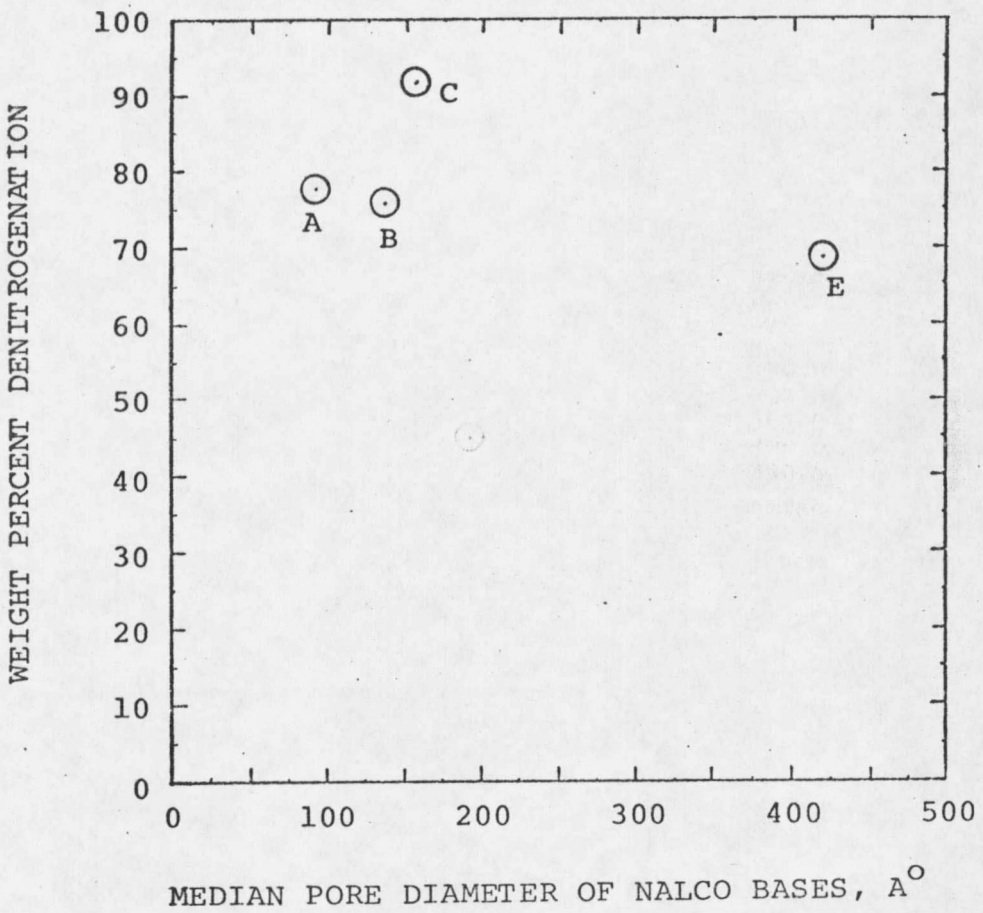


FIGURE 18. EFFECT OF MEDIAN PORE DIAMETER ON THE DENITROGENATION

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

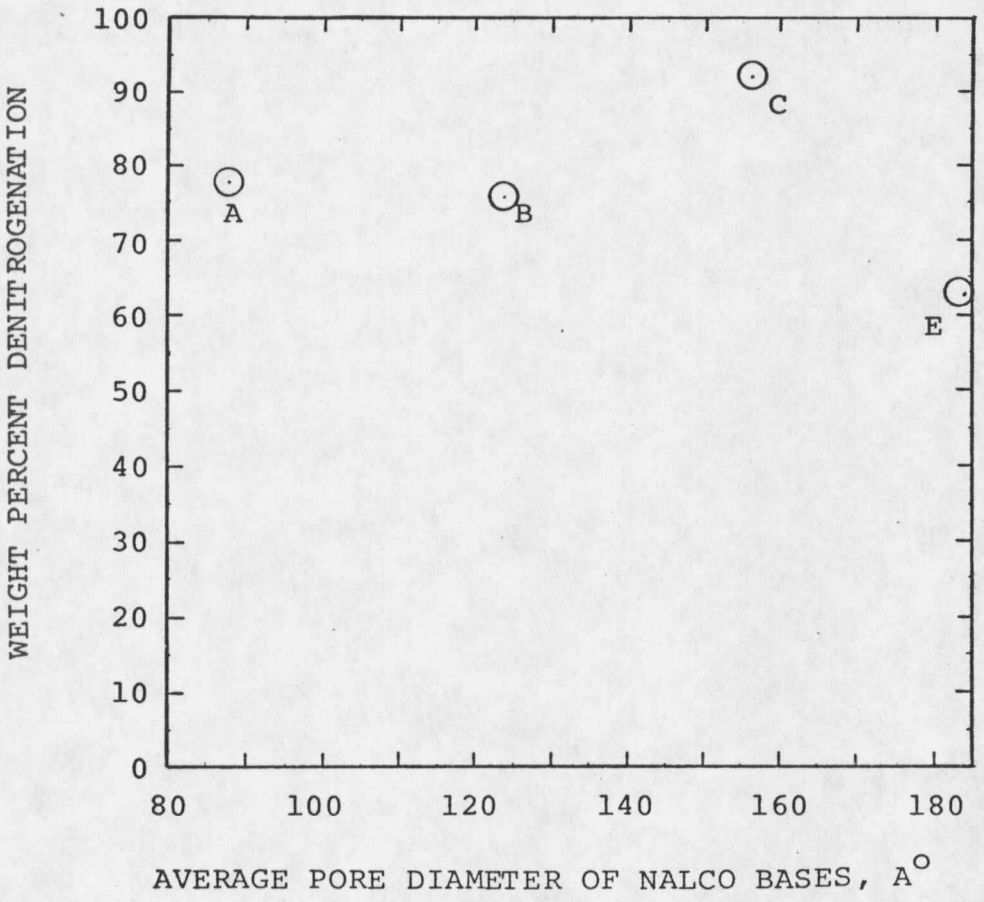


FIGURE 19. EFFECT OF AVERAGE PORE DIAMETER ON THE DENITROGENATION

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

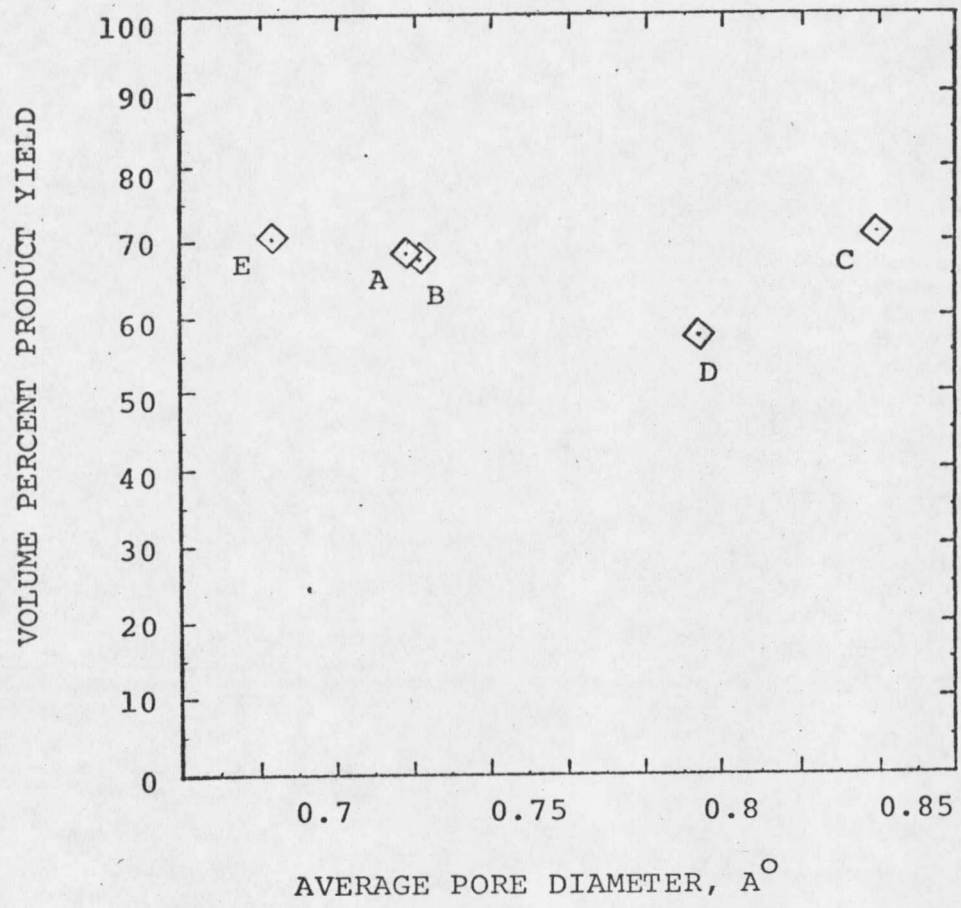


FIGURE 20. EFFECT OF PORE VOLUME ON THE DENITROGENATION

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

with 6% MoO_3. In general, the Nalco B based or Nalco E based catalyst was not as good as the Nalco C based catalyst with the same amount of MoO_3

The next twenty catalysts were fabricated using Nalco C base to evaluate the effect of metal concentration on the catalytic performance (Table IX).
The first four catalysts in the table contained the same amount of MoO_3 (0.04906 gm per gm base). The percentage varied depending upon the additional metals impregnated. It was originally intended to make the second four catalysts contain approximately twice the amount of MoO_3 as the first group. In a similar fashion efforts were made to make the third four catalysts have three times as much MoO_3 as the first group and so on.

Run 9, Run 19, Run 24, Run 37 and Run 33 along with Run 12 used Nalco C based catalysts. The average percent denitrogenations obtained for the first two hours of each run were plotted against the MoO_3 concentration in Figure 21. The maximum denitrogenation was realized at a MoO_3 concentration of around 12%.
Although each catalyst used for Run 29, Run 20, Run 25, Run 38, and Run 34 contained a slightly different amount

Table IX

COMPOSITION AND PERFORMANCE OF MSU KN C SERIES CATALYST

Cat No.	Run No.	MoO_3 %	MoO_3 gm/gm base	NiO %	NiO gm/gm base	CoO %	CoO gm/gm base	WO_3 %	WO_3 gm/gm base	% DN	% DS	Yield
C9	28	4.68	(0.04906)	0.	(0.)	0.	(0.)	0.	(0.)	86.8	-	61.7
C10	29	4.62	(0.04906)	1.32	(0.01406)	0.	(0.)	0.	(0.)	92.9	16.1	68.7
C11	31	4.26	(0.04906)	1.22	(0.01406)	7.76	(0.08948)	0.	(0.)	87.7	-	57.9
C12	32	3.78	(0.04906)	1.08	(0.01406)	6.90	(0.08948)	11.16	(0.14480)	93.2	36.5	69.3
C1	19	8.32	(0.09080)	0.	(0.)	0.	(0.)	0.	(0.)	86.5	-	63.8
C2	20	7.99	(0.09080)	3.98	(0.04520)	0.	(0.)	0.	(0.)	86.2	66.5	76.3
C3	21	7.40	(0.09080)	3.69	(0.04520)	7.42	(0.09110)	0.	(0.)	82.0	65.1	67.1
C4	22	6.45	(0.09080)	3.22	(0.04520)	6.46	(0.09110)	12.89	(0.18160)	88.3	-	67.1
C5	24	12.08	(0.13744)	0.	(0.)	0.	(0.)	0.	(0.)	100.0	38.3	47.9
C6	25	11.92	(0.13744)	1.34	(0.01547)	0.	(0.)	0.	(0.)	97.2	78.7	67.1
C7	26	11.24	(0.13744)	1.27	(0.01547)	5.74	(0.07020)	0.	(0.)	88.3	61.1	63.2
C8	27	10.02	(0.13744)	1.13	(0.01547)	5.12	(0.07020)	10.82	(0.14836)	91.3	84.1	71.2
C17	37	15.36	(0.18147)	0.	(0.)	0.	(0.)	0.	(0.)	92.0	-	63.8
C18	38	15.11	(0.18147)	1.66	(0.01994)	0.	(0.)	0.	(0.)	94.4	86.7	69.6
C19	39	14.16	(0.18147)	1.56	(0.01994)	6.23	(0.07985)	0.	(0.)	94.4	75.0	57.0
C20	40	12.72	(0.18147)	1.40	(0.01994)	5.60	(0.07985)	10.20	(0.14560)	92.8	89.0	60.5
C13	33	17.77	(0.21616)	0.	(0.)	0.	(0.)	0.	(0.)	94.7	65.4	52.5
C14	34	17.66	(0.21616)	0.64	(0.00777)	0.	(0.)	0.	(0.)	86.6	-	56.3
C15	35	16.88	(0.21616)	0.61	(0.00777)	4.42	(0.05653)	0.	(0.)	86.6	36.7	60.0
C16	36	15.30	(0.21616)	0.55	(0.00777)	4.00	(0.05653)	9.39	(0.12376)	90.7	76.0	57.9

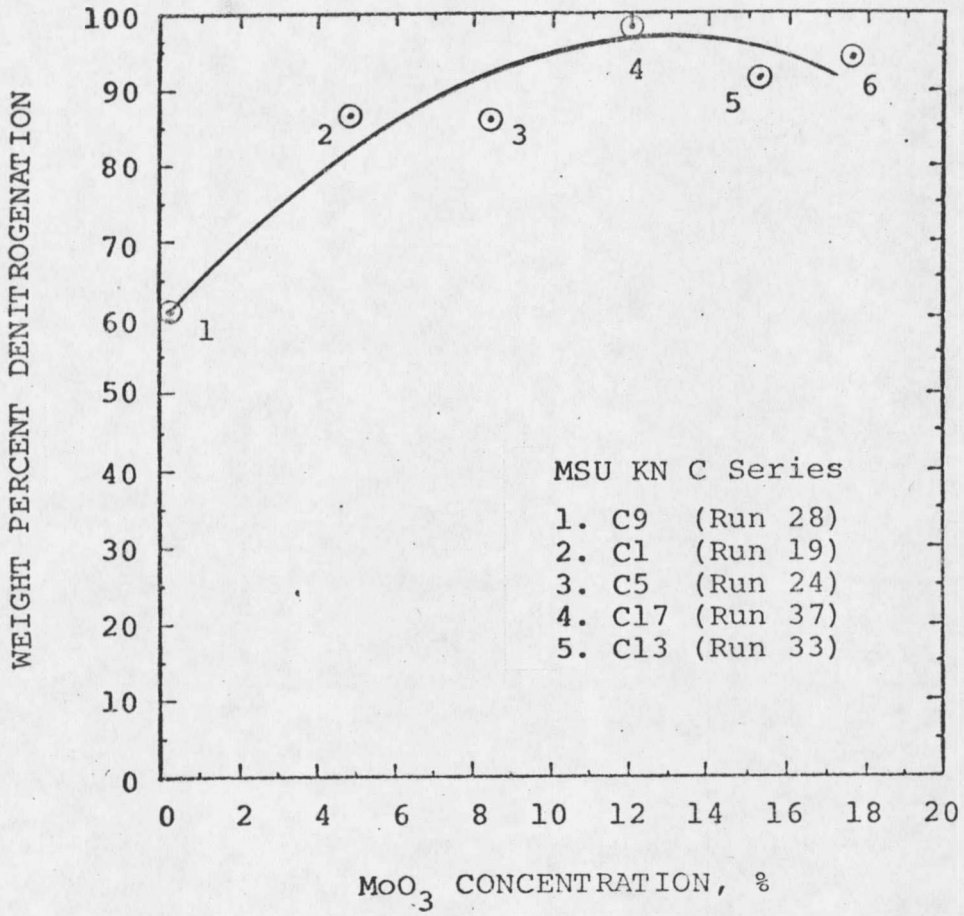


FIGURE 21. EFFECT OF MoO_3 CONCENTRATION ON THE DENITROGENATION

of NiO, Figure 22 was prepared to see if this small addition of NiO could enhance the catalytic performance at different MoO_3 concentrations. The percent denitrogenation remained almost the same.

The effect of the CoO presence in the previous group of catalysts on the percent denitrogenation was shown in Figure 23 based on the results obtained from Run 31, Run 21, Run 26, Run 39, and Run 35. Again, no significant change was noticed. The third additional metal, WO_3, however, displayed a tendency to level the difference in denitrogenation as shown in Figure 24 based on the results of Run 32, Run 22, Run 27, Run 40, and Run 36.

The product yields obtained from Run 28, Run 19, Run 24, Run 37, Run 33, and Run 15 were plotted against the MoO_3 concentrations in Figure 25. No significant correlation was found.

The linear regression analysis indicated that there was no significant correlation between the percent denitrogenation and product yield (R^2=0.1).

Regeneration of The Best Catalyst

The MSU KN C5 catalyst which was previously tested in Run 24 and proven best among the catalysts made by the

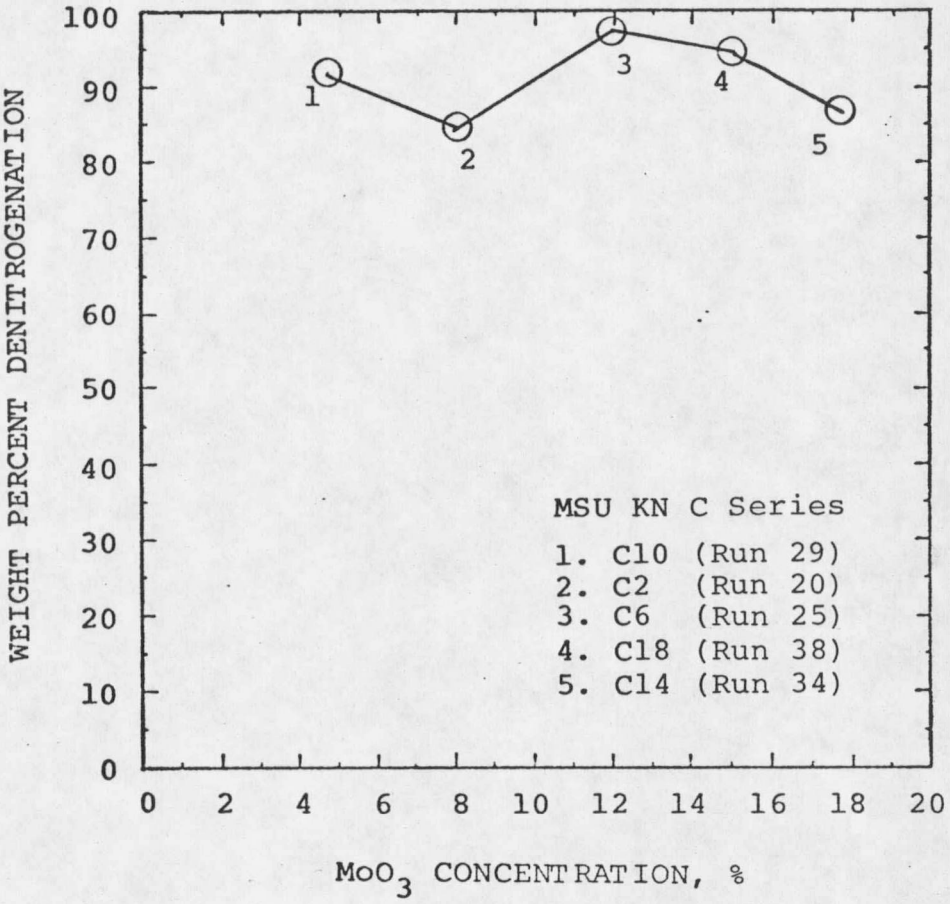


FIGURE 22. EFFECT OF MoO_3 CONCENTRATION ON THE DENITROGENATION IN THE PRESENCE OF NiO.

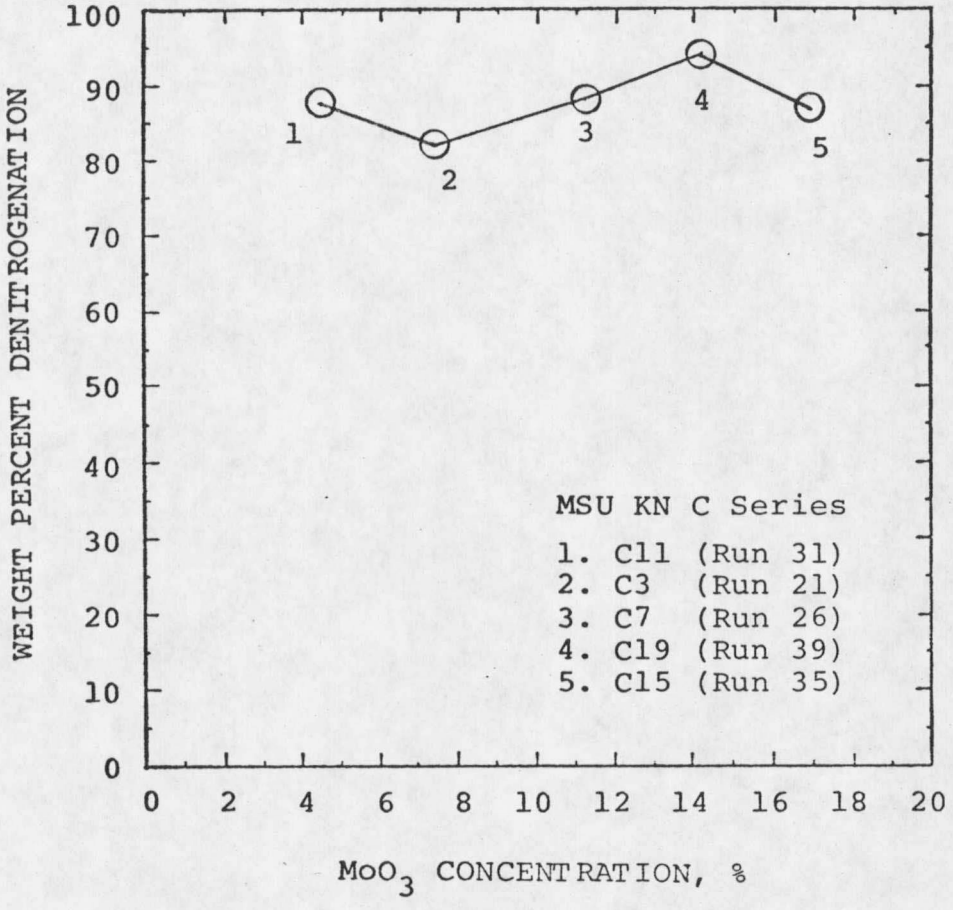


FIGURE 23. EFFECT OF MoO_3 CONCENTRATION ON THE DENITROGENATION IN THE PRESENCE OF NiO and CoO.

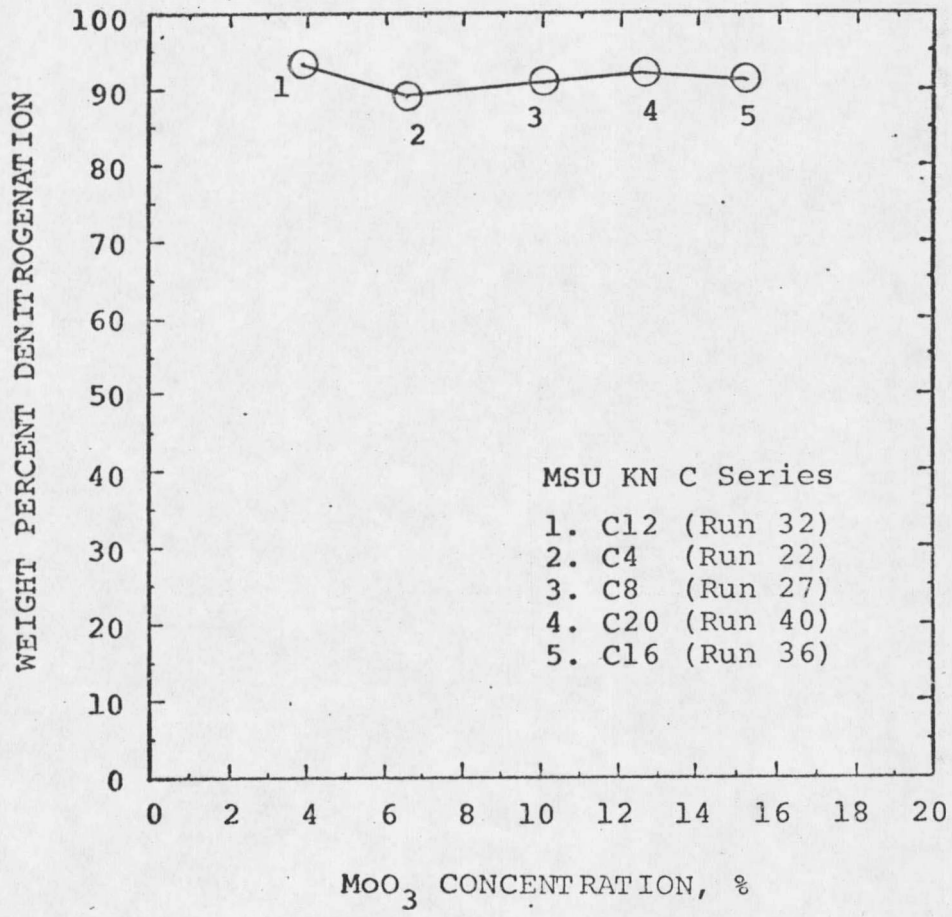


FIGURE 24. EFFECT OF MoO_3 CONCENTRATION ON THE DENITROGENATION IN THE PRESENCE OF NiO, CoO, and WO_3.

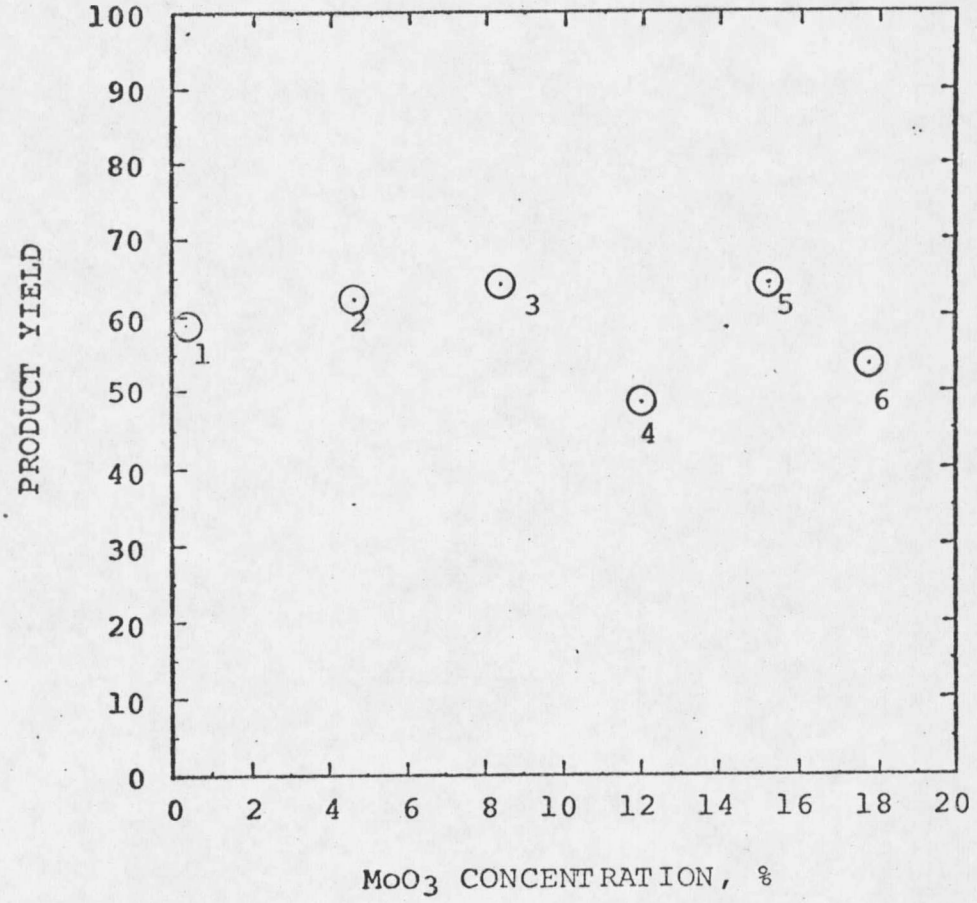


FIGURE 25. EFFECT OF MoO_3 CONCENTRATION ON THE PRODUCT YIELD

MSU KN C Series

1. Nalco C Base (Run 12)
2. C9 (Run 28)
3. C1 (Run 19)
4. C5 (Run 24)
5. C17 (Run 37)
6. C13 (Run 33)

author was chosen for a long-run evaluation of its durability and regeneration potentiality. A summary of operating conditions and analytical results is shown in the data sheet of Run 30.

The first part of Run 30 was carried out for 6 hours. The reactor was cooled following a brief washing with toluene. The percent nitrogen removal from the products was plotted against time on stream in Figure 26. Performance of the virgin catalyst was excellent at an average denitrogenation of 95% for the first 6 hours. An additional 4-hour run was made and washed with the toluene again. Subsequent nitrogen and sulfur removal were added to Figure 26. The percent denitrogenation curve did not follow the predicted curve A but improved significantly, creating a new curve B. This is primarily due to intermediate solvent washing. A part of the carbonaceous deposit must have been removed, reopening the pores. The third part of Run 30 was carried out for 6 more hours to make a total of 16 hours. The results of the nitrogen analyses showed another jump in Figure 26 as previously experienced (curve C). If the experiment had been carried out without interruptions, a curve A

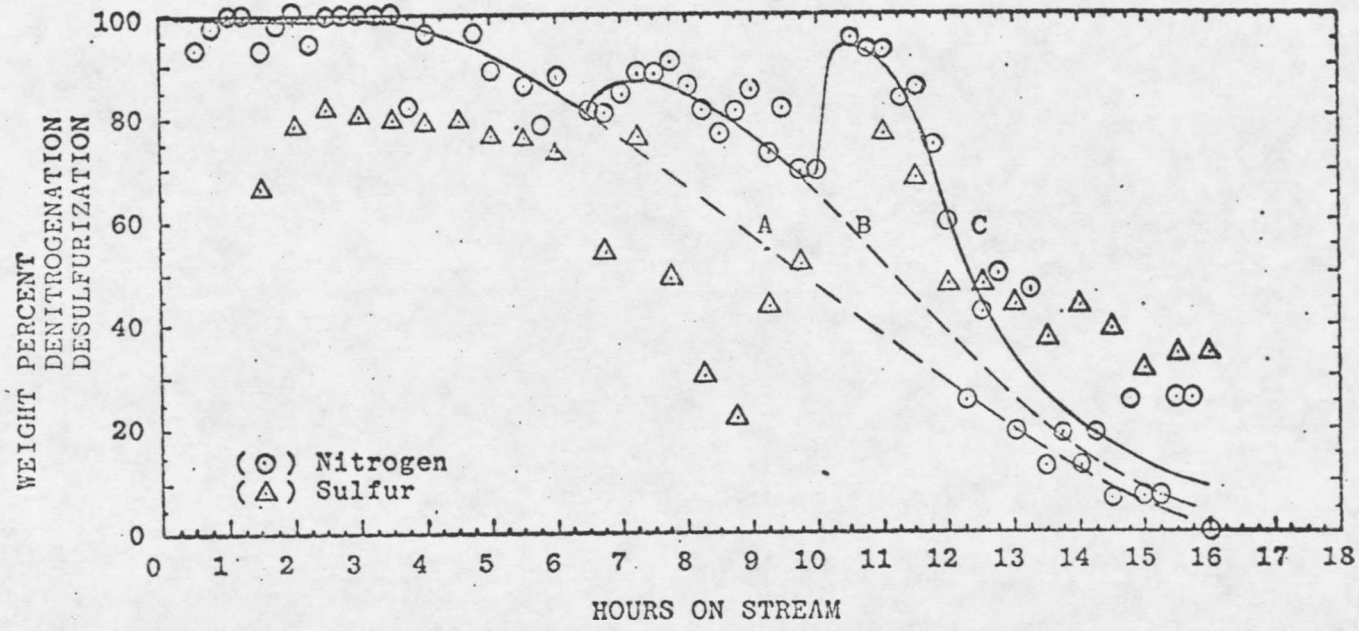


FIGURE 26. PERFORMANCE OF MSU KN C5 CATALYST (VIRGIN) IN DENITROGENATION AND DESULFURIZATION

would have been obtained. Figure 26 also showed the percent desulfurization. The sulfur removal was good at 80% for the first 6 hours. The second part of the desulfurization curve seemed erratic but the third part of the desulfurization curve showed a good pattern.

The first thermal regeneration for this catalyst was made by heating the in situ catalyst in the reactor. Three different gas mixtures were used in sequence; (1) $1.5\%O_2$-$98.5\%N_2$ gas mixture, (2) $5\%O_2$-$95\%N_2$ gas mixture, and (3) air. A regeneration temperature of $600^{\circ}C$ was adopted as a standard after a number of trials starting from $500^{\circ}C$ at $50^{\circ}C$ intervals (Figure 9).

The regeneration, actually burnoff of carbonaceous deposit, was carried out using $1.5\%O_2$-$98.5\%N_2$ gas mixture at high temperature for two hours and followed by $5\%O_2$-$95\%N_2$ gas mixture for another two hours. Then the air was connected to complete the burnoff for a 24-hour period. The temperature remained almost constant at $600^{\circ}C$ throughout the burnoff process. No incidents of sharp temperature increase as previously experienced occurred. A minor temperature fluctuation in the magnitude of $\pm 20^{\circ}C$ was mostly attributed to the electric

furnace controller cycle rather than to the combustion.

The catalyst was inspected under the microscope to confirm the completeness of burnoff. When no trace of carbon was observed, the catalyst was presulfided for Run 30R1.

Run 30R1 was carried out for 14 hours without interruption using the first regenerated catalyst. The operating conditions and analytical results are presented in the data sheet of Run 30R1. Performance in nitrogen removal was satisfactory but was not as good as the virgin. The exhausted catalyst was regenerated for the second time in the similar fashion and used for the next 16-hour run (Run 30R2).

The overall performance of the MSU KN C5 catalyst is presented in Figure 27 for denitrogenation and Figure 28 for desulfurization. The second-regenerated catalyst performed better than the first-regenerated but was not as good as the virgin. A mathematical model was developed to predict the percent denitrogenation as a function of time on stream. The best fit appeared to take a form of the Logistic growth law(66) as follows;

$$\%DN = 100. - \frac{100.}{(1. + A*B**T)}$$

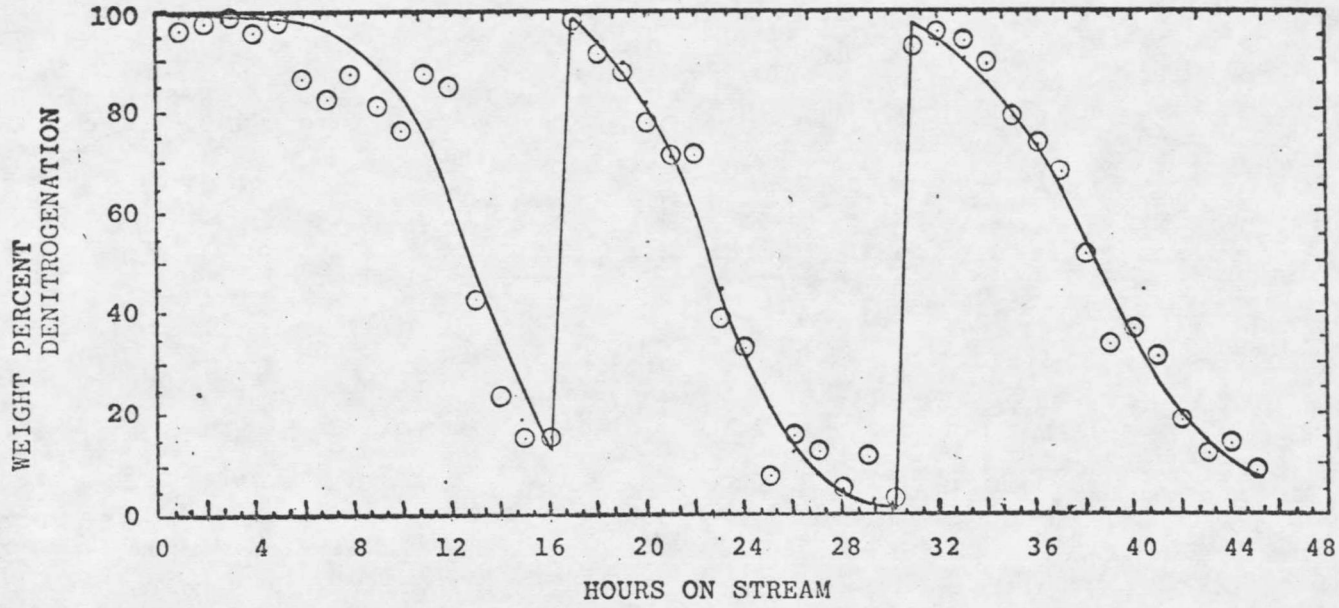


FIGURE 27. OVERALL PERFORMANCE OF MSU KN C5 CATALYST IN DENITROGENATION FROM THE VIRGIN THROUGH THE SECOND-REGENERATED

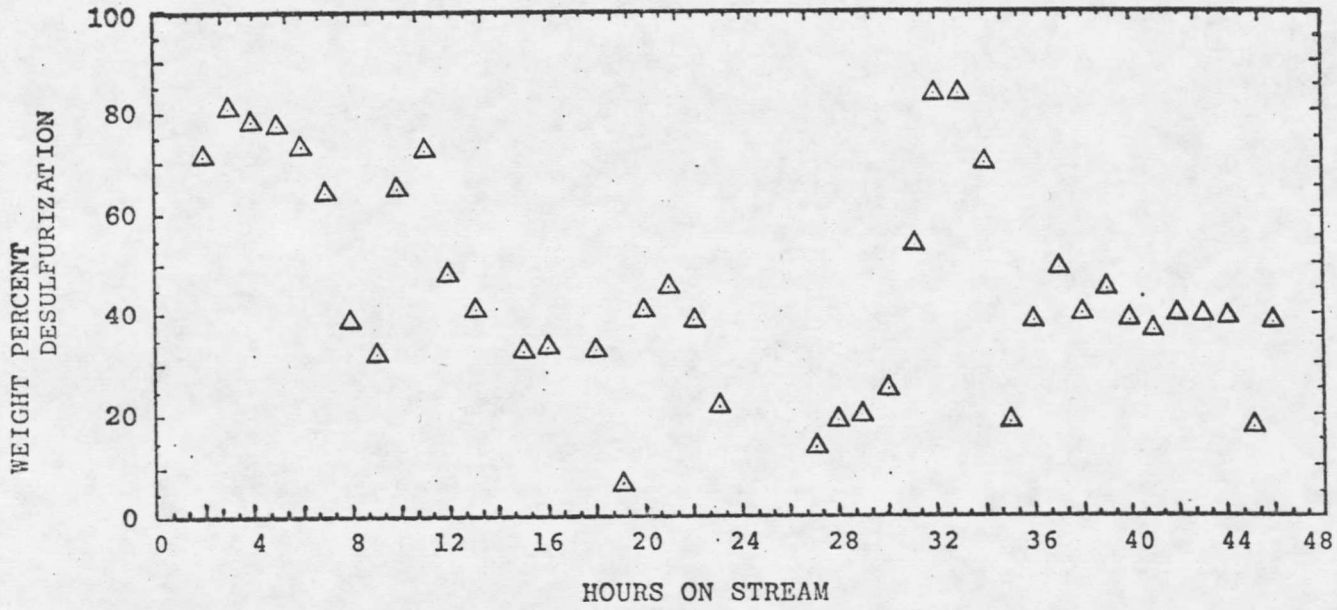


FIGURE 28. OVERALL PERFORMANCE OF MSU KN C5 CATALYST IN DESULFURIZATION FROM THE VIRGIN THROUGH THE SECOND-REGENERATED

where A and B = constants

T = time in hour

A computer program was written to minimize the objective function(the sum of the square of the differences between the experimental values and calculated values obtained using the above model) using the complex method of Box. The computer calculated constants for each run are presented below and shown as solid lines in Figure 27.

	Constants	
Catalyst	A	B
Virgin	3670.	0.53
1st-Regenerated	41.6	0.57
2nd-Regenerated	34.9	0.66

The ASTM D-86 atmospheric distillation was conducted on the selected composite sample of the products. A typical characteristic of the SRL, raw feed solution, composite product sample(Run 18) is shown in Figure 29 along with the boiling point of tetralin under the Bozeman barometric pressure of 640 mm Hg.

The solid SRL showed some distillate (approximately 30%) at 665^{o}F. The raw feed solution showed some dissolution,

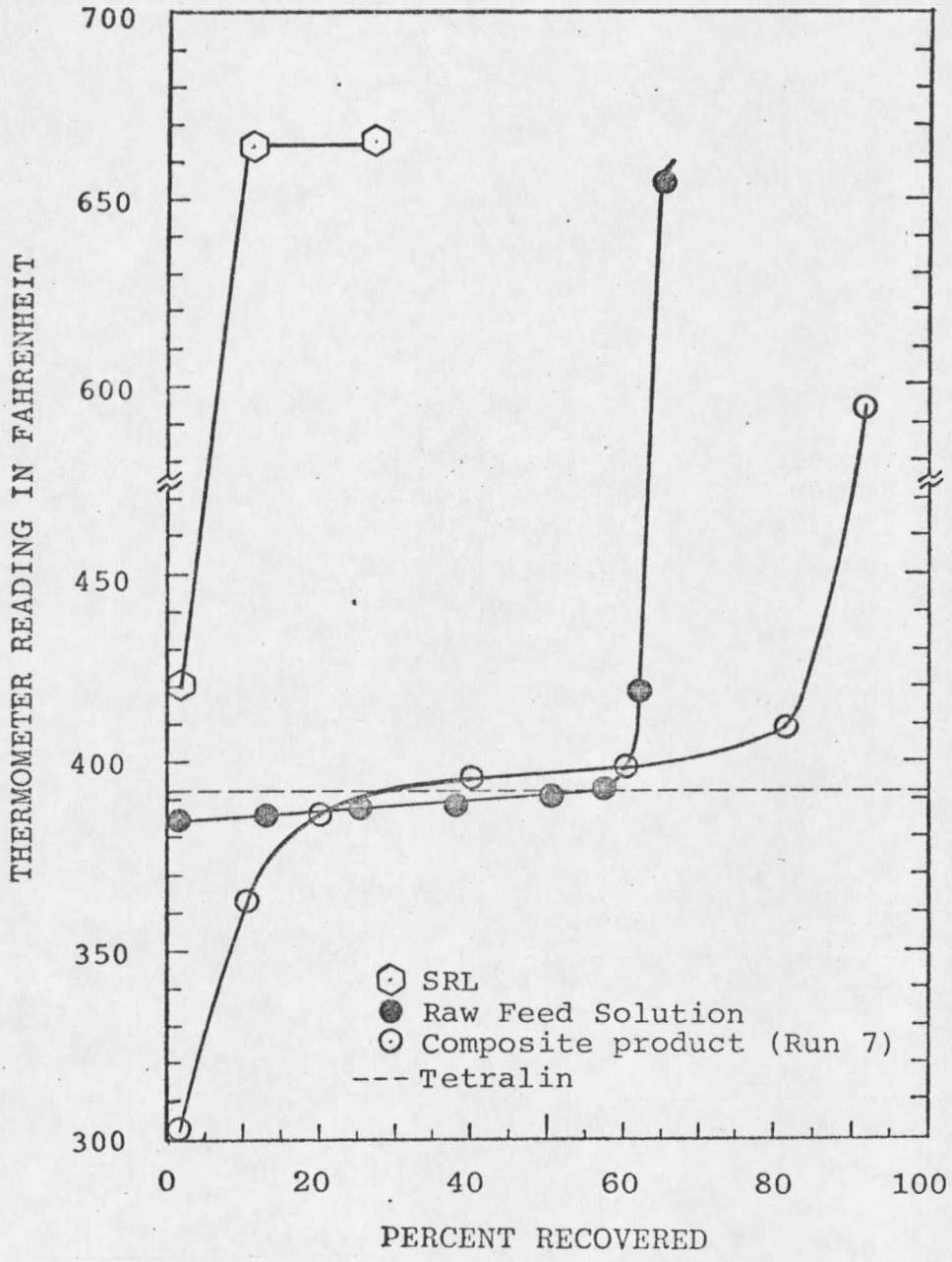


FIGURE 29. DISTILLATION DATA OF RUN 7 COMPOSITE PRODUCT SAMPLE AND RAW FEED SOLUTION ALONG WITH THE SRL.

while the product displayed dissolution and hydrocracking of the SRL to some extent.

Inspection of the Reactor Content

A former fellow researcher experienced plugups frequently after about 2 hours of run but the author has never experienced this, although the pressure increased up to 2100 psig during Run 1. In an effort to see what is happening within the reactor, the entire content of the reactor was drained on the countertop in an orderly manner immediately after each run. This was a departure from the previous practice which called for flushing of the reactor with motor oil and cooling prior to drain. The immediate dump while the reactor was hot gave an opportunity to better understand the liquid feed distribution and to analyze the exhausted catalyst for nitrogen content. Some of the significant observations were;

(1) The ¼-inch Denstone support of the preheating zone (top of the reactor) was well coated with the concentrated feed, indicating a possibility of plugups.

(2) Visual inspection of the entire spectrum indicated that the feed solution might have been partially vaporized.

(3) The heavy layers of carbonaceous deposit penetrated deeper down into the reactor, as the operating hours increased.

(4) A sign of short-circuiting within the reactor was noticed but it was considered to be insignificant.

The exhausted catalyst and supports were separated into several sections for nitrogen analysis (Run 10). The analytical results were summarized as follows;

Sections	Wt % N
¼-inch Denstone	0.94
1/32-inch catalyst, top	0.54
1/8-inch Denstone, top	1.35
1/32-inch catalyst, bottom	0.30
1/8-inch Denstone, bottom	0.45
Carbonaceous deposit, reactor wall	0.75

The reactor holdup, defined as the difference in the total weight of the reactor before and after a run, varied from 15 gm to 25 gm. Based on the 2-hour run, they were approximately equivalent to 12.5% (Run 12) to 20.8% (Run 14) of the total feed solution. This is commonly referred to as the residual holdup. The free draining holdup varied from 8 gm (Run 38) to 18 gm

(Run 41).

Structure of Nalco Base Catalysts

An effort was made to predict the optimum metal oxides and the subsequent pore diameter. Calculations indicated that metal impregnation on the Nalco base may reduce the original average pore diameter by 16% to 38%, depending upon the type of base used.

The key assumptions that the calculations were based on are as follows(64);

(1) The surface area was covered with a monolayer of molecules for each metal compound impregnated.

(2) The constituent molecule assumed the covalent bond radius.

(3) The ammonium molybdate hexahydrate assumed a structure of paramolybdate ion (octahydra) during the incipient wet stage. The molybdenum oxide is formed by calcining the ammonium molybdate, creating holes on the monolayer due to escaping ammonia and combined water.

(4) The ammonium metatungstate assumed a 12-tungstoheteropoly anion, $(Mo_7O_{24})^{6-}$, in solution.

(5) The pore assumed a cylindrical shape.

The maximum metal loading for a monolayer coverage has been calculated and summarized in Table X. For example, one of the best MSU catalysts(46,47) had an approximate MoO_3 content of 12%. This is equivalent to 0.176 gm of MoO_3 per gm base (Nalco C) which is sufficient to cover as a monolayer over the reported surface area of 214 m^2. Further addition of MoO_3 will theoretically not increase any effective surface area but rather reduce the pore diameter. Although it is highly conceptual, it agreed well with the experimental results and was a facilitation in predicting the optimum metal loading.

Some Nalco-based catalysts were inspected under the electron microscope (MSU Veterinary Research Laboratory). The Nalco C base prior to any metal impregnation is shown in Figure 30-A as compared with the MSU KN C8 catalyst with 10.02% MoO_3, 1.13% NiO, 5.12% CoO, and 10.82% WO_3 in Fig. 30-B. All pictures were taken with a magnification of 30,000 times so that one millimeter on the pictures is equivalent to 320 A (1A=one ten-billionth of a meter). The pictures clearly showed that the basic granules constituting the Nalco base assumed a spherical shape, which characterized the hydrodynamic behavior of

Table X

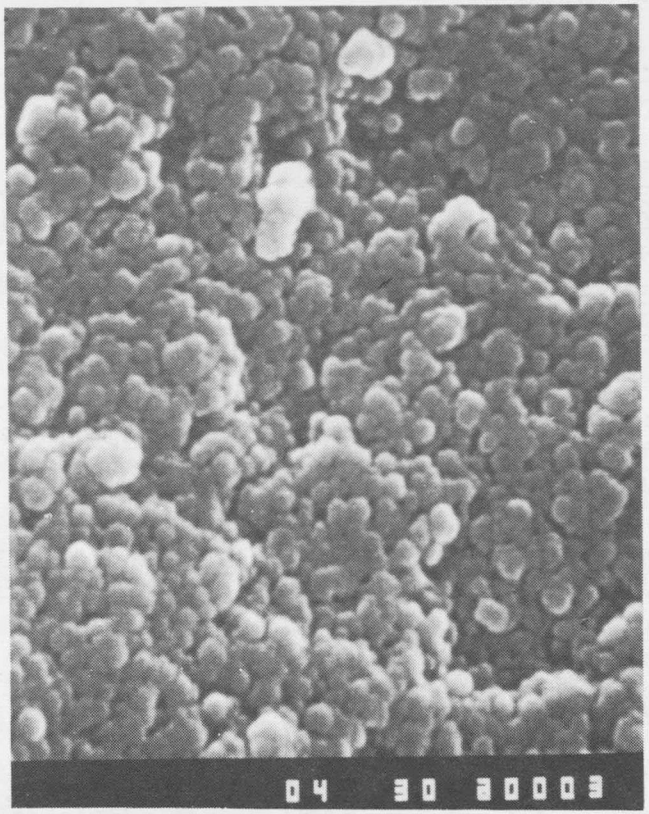
CALCULATED MAXIMUM IMPREGNATION OF METAL OXIDES AND AVERAGE PORE DIAMETER

	Nalco A	Nalco B	Nalco C**	Nalco D	Nalco E
Base, gm	1.0	1.0	1.0	1.0	1.0
MoO_3, gm	0.268	0.193	0.178	0.175	0.122
NiO , gm	0.167	0.12	0.111	0.109	0.076
CoO , gm	0.212	0.152	0.141	0.138	0.0965
WO_3 , gm	0.553	0.397	0.366	0.361	0.252
Avg Pore Dia., ^{o}A (1)*	89	124	156	150	186
(2)	56	90	122	116	152

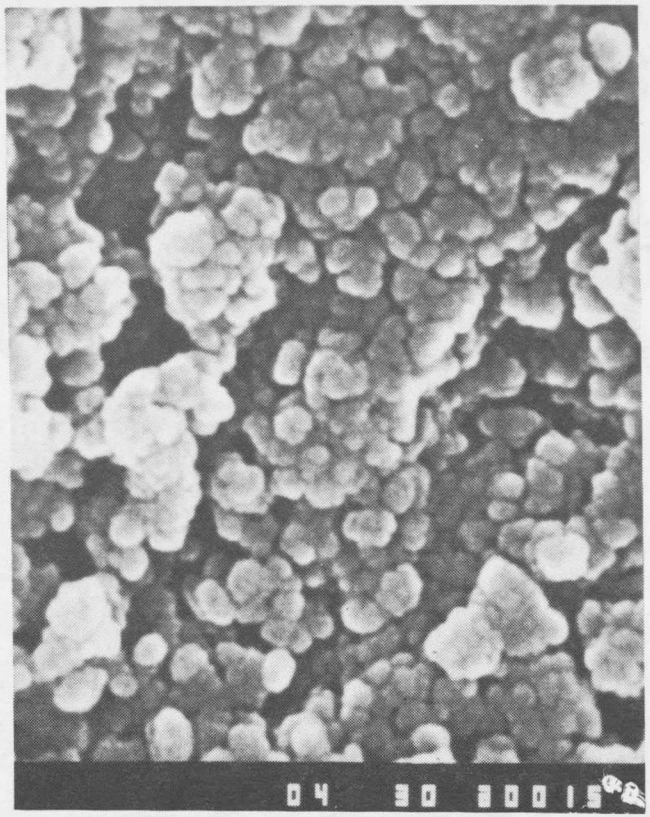
* (1) Average pore diameter of the original base
(2) Average pore diameter after impregnating 4 metal oxides. This is calculated based upon an assumption of monolayer coverage.

** One of the best MSU catalyst has an approximate composition as follows;

	Percent	Weight, gm
Base, Nalco C	68.0	1.0
MoO_3	12.0	0.176
NiO	3.0	0.044
CoO	7.0	0.103
WO_3	10.0	0.147

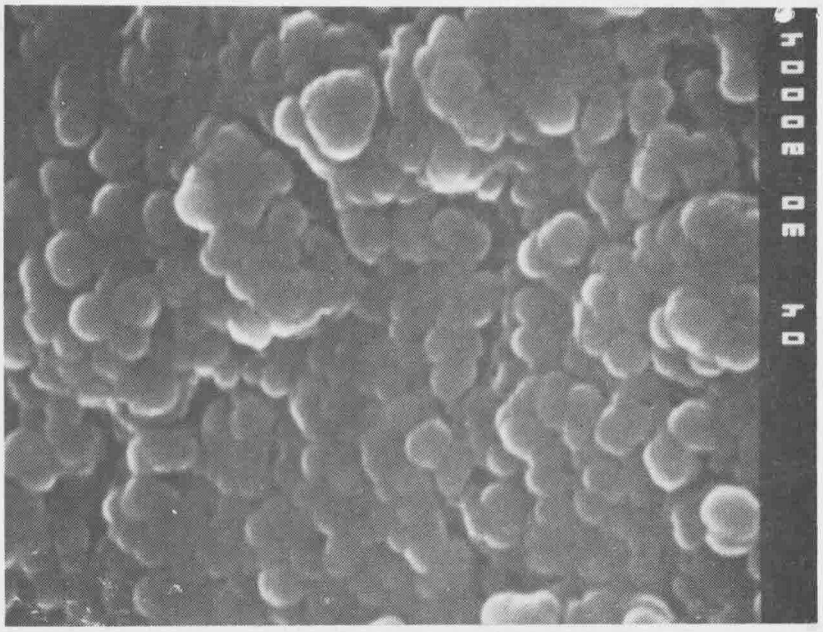


(A)

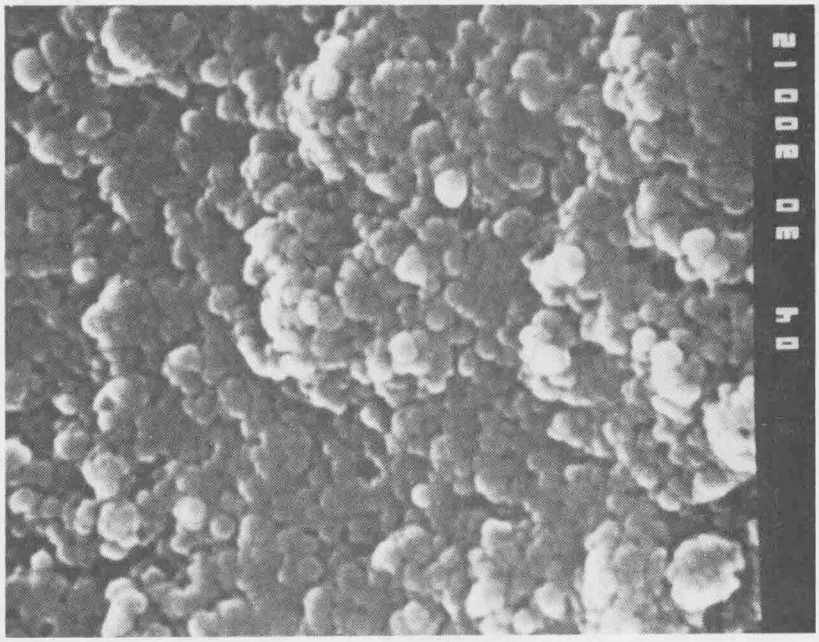


(B)

FIGURE 30. SCANNING ELECTRON PHOTOMICROGRAPHS:
(A) NALCO BASE C, 2%SiO_2;98%Al_2O_3 (B) MSU KN C8 CATALYST WITH 10.02% MoO_3, 1.13%NiO, 5.12%CoO, and 10.82%WO_3 on NALCO BASE C.
(C) MSU KN C5 CATALYST WITH 12.08%MoO_3 ON NALCO BASE C
(D) MSU KN C5 CATALYST AFTER SECOND REGENERATION



(D)



(C)

the fluids within the catalyst.

If the spheres were stacked together either in "hexagonal closest-packing" or in "cubic closest-packing", the maximum diameter of a ball for a surface area of 215 m^2 was calculated to be approximately 100 A. Therefore, a lump of 1 millimeter in diameter as measured on the picture contains about 24 balls. Figure 30-C & D showed the MSU KN C5 catalyst(new) in contrast to the second-regenerated. The granules of the second-regenerated catalyst appeared to be larger, indicating a possible sintering. Otherwise,it could be a sampling error.

Rough Nitrogen Balance

A rough balance was made on nitrogen using the data partially collected from Run 10 and Run 26. A summary of the calculations was presented in Table XI.
It appeared that the majority of nitrogen (68.5%) was gasified and trapped in the impingers with concentrated sulfuric acid. Approximately 20% of the total nitrogen remained within the reactor.

Table XI

ROUGH NITROGEN BALANCE

(1) Input		
Total feed volume: 120 ml Total feed weight: 120 ml(0.9gm/ml)=108gm Nitrogen(N) concentration: 0.27% N: 108 gm (0.0027)=	0.2916 gm	
(2) Output		
(a) Total product (Run 26) volume: 76 ml Total product weight: 76 ml (0.98gm/ml)=74.48 gm N concentration: 0.34% N: 74.48 gm (0.0034)=	0.0253 gm	8.7%
(b) Reactor holdup weight: 15 gm N concentration: 15 gm (0.003967)=	0.0595 gm	20.4%
(c) Impinger Catch Total sulfuric acid: 113 ml N concentration: 0.096% N: 113 ml (1.841 gm/ml)0.00096=	0.1997 gm	68.5%
(d) N unexplained (difference)	0.0071 gm	2.4%
Total	0.2916 gm	100.0%

CONCLUSION

1. The ratio of SRL to tetralin of 1:2 and pretreatment with a centrifuge were necessary to reduce the pumping problem due to suspended partcles.
2. Increased MoO_3 concentration of up to 12% by weight on Nalco C base favored better denitrogenation. This amount of MoO_3 was also theoretically proved enough to cover a surface area of 215 m^2 per gm base as a monolayer. Excessive MoO_3 would not increase any effective surface area but only reduce the pore diameter.
3. The effect of additional metal promoters, NiO and CoO, on nitrogen removal was not significant when the metal impregnation was in the order of MoO_3, NiO, CoO, and WO_3. If the sequence of metal impregnation was altered, the presence of these promoters could have given a synergistic effect.
4. Presence of tungsten trioxide is favorable for nitrogen removal until a total of MoO_3 and WO_3 reached 12%.
5. The Nalco blank carriers, either in pure Al_2O_3 or in a combination of SiO_2+Al_2O_3, also demonstrated the

capacity of denitrogenation and desulfurization to some extent. An optimum combination of pore volume and surface area made a good catalyst carrier.

6. MSU KN C5 with 12.08% MoO_3 on Nalco C base (2% SiO_2+ 98% Al_2O_3) demonstrated that the catalytic performance could be fully restored in two-consecutive thermal regeneration.
7. Among the distillation data of few selected composite samples, MSU KN A1 with 8.42% MoO_3, 1.29% NiO, 4.00% CoO, and 10.04% WO_3 gave a most desirable pattern. The effect of additional metals on the hydrocracking appears to be significant.
8. Rough nitrogen balance showed that the majority of nitrogen compound (68.5%) was gasified and approximately 20.4% remained in the carbonaceous deposit within the reactor after the two-hour operation.
9. The best catalyst for desulfurization was MSU KN C20 having 12.72% MoO_3, 1.40% NiO, 5.60% CoO, and 10.20% WO_3.

RECOMMENDATION FOR FUTURE RESEARCH

1. The existing pump should be modified so that the raw feed solution can be pumped without any centrifuge pretreatment.
2. The more severe operating conditions, a reactor pressure in the range of 1500 to 3000 psig and a reaction temperature in the range of 500 to 840^{o}F, should be tested to improve the hydrogenation of SRL dissolved in tetralin.
3. Further study should be carried out in establishing mathematical and geometrical models to explore the catalytic reaction with the aids of electronic microscope and X-ray deflection analysis.

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APPENDIX A

DISTILLATION DATA

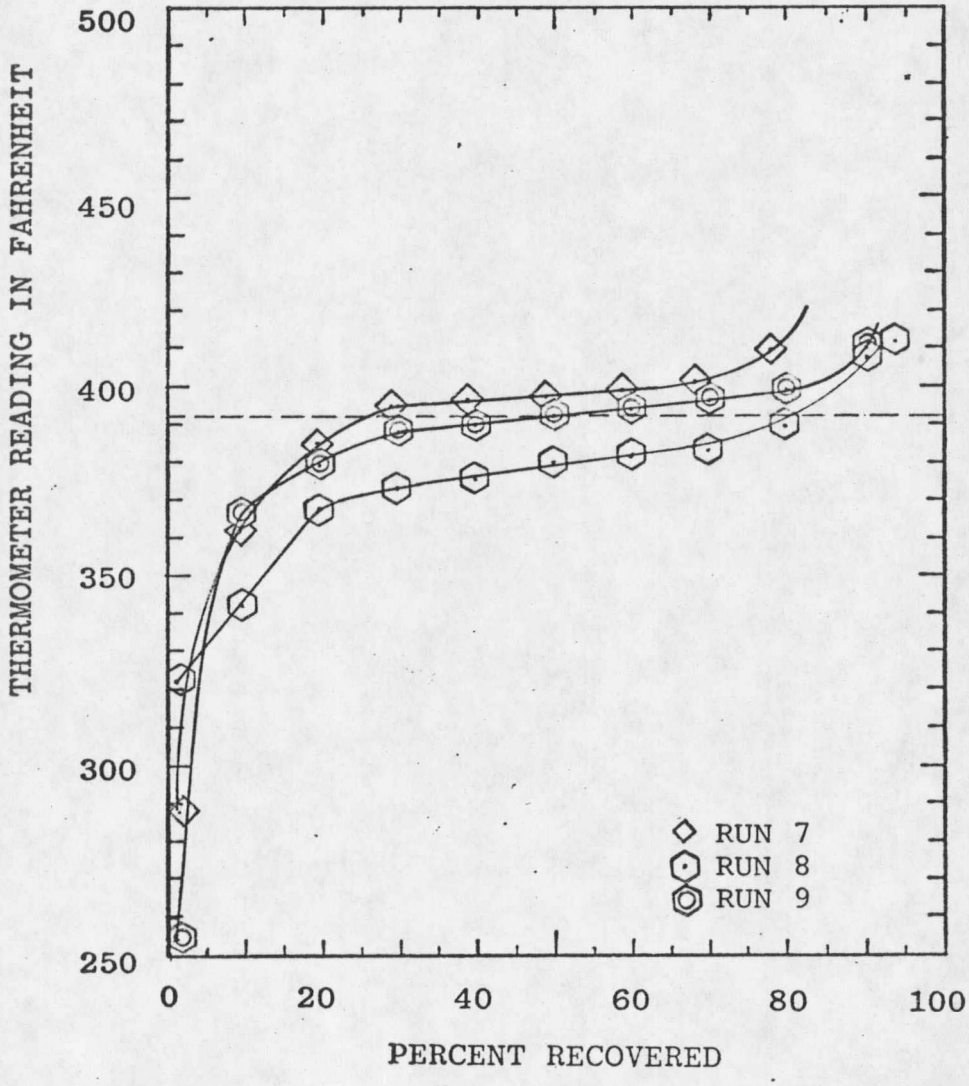


FIGURE 31. DISTILLATION DATA OF COMPOSITE PRODUCTS FROM RUN 7, RUN 8, AND RUN 9.

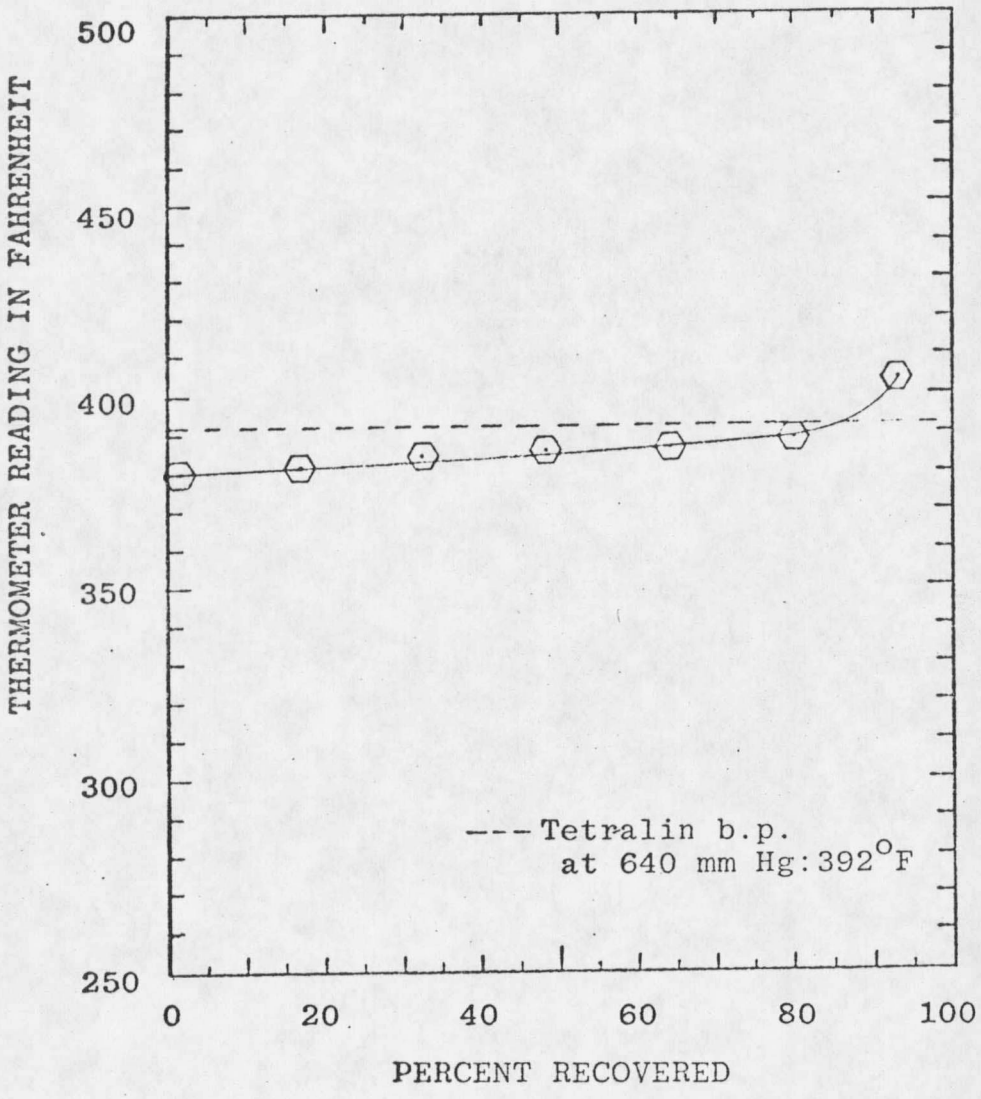


FIGURE 32. DISTILLATION DATA OF RUN 15
COMPOSITE PRODUCTS

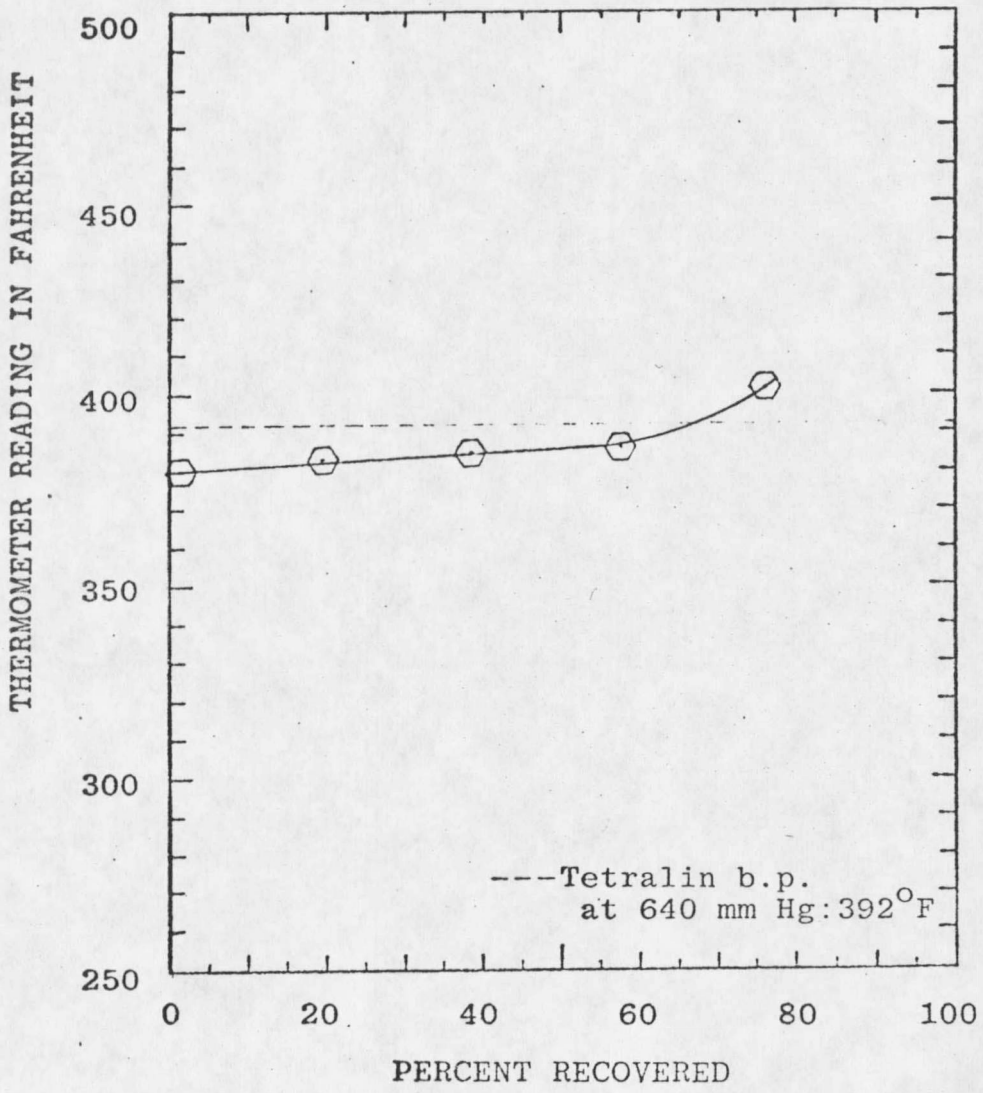


FIGURE 33. DISTILLATION DATA OF RUN 16
COMPOSITE PRODUCTS

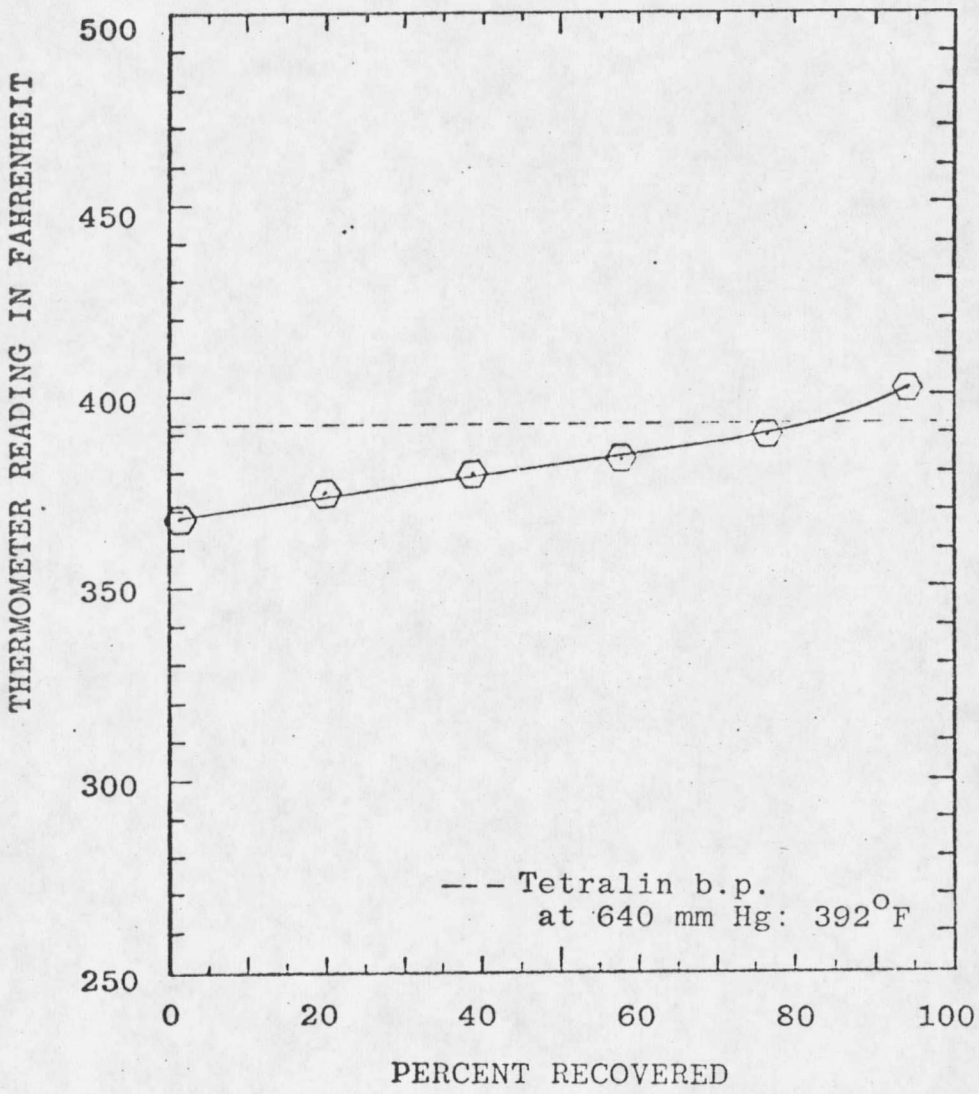


FIGURE 34. DISTILLATION DATA OF RUN 17
COMPOSITE PRODUCTS

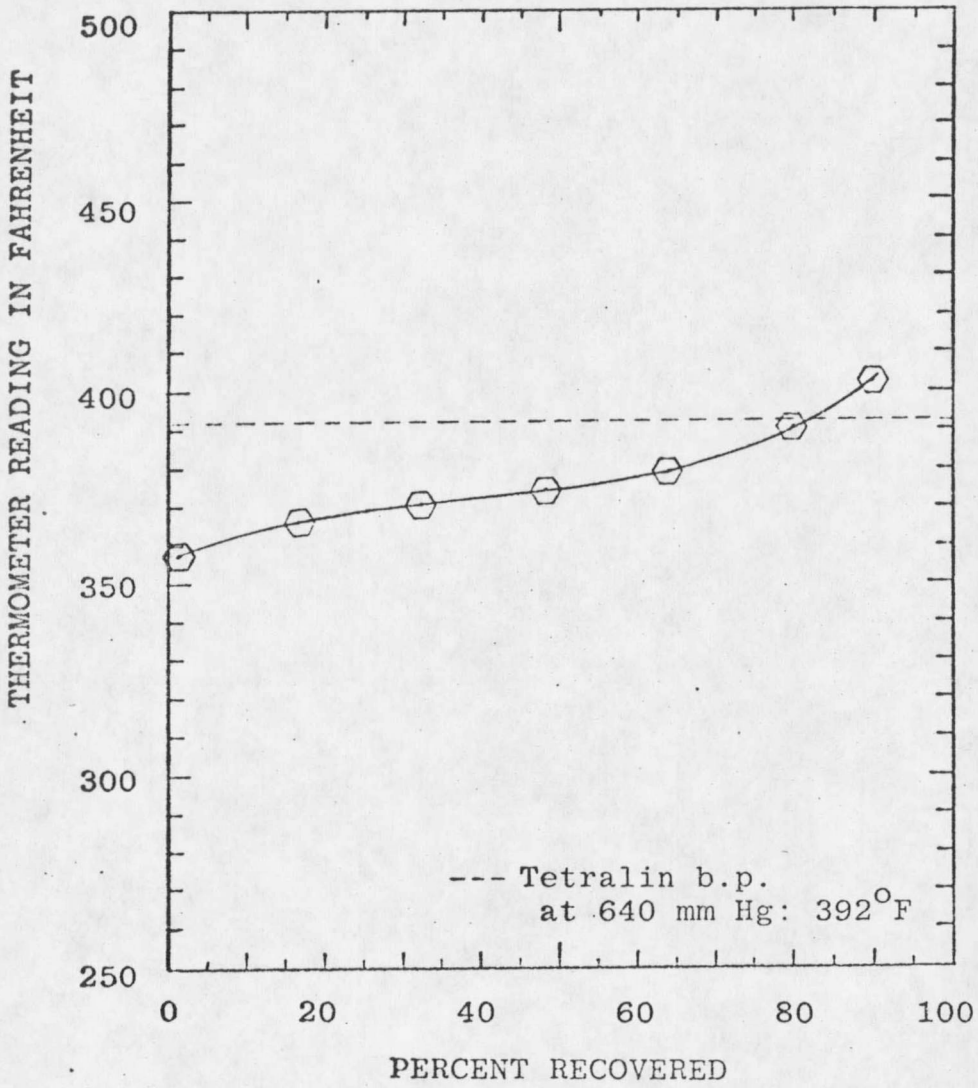


FIGURE 35. DISTILLATION DATA OF RUN 18
COMPOSITE PRODUCTS

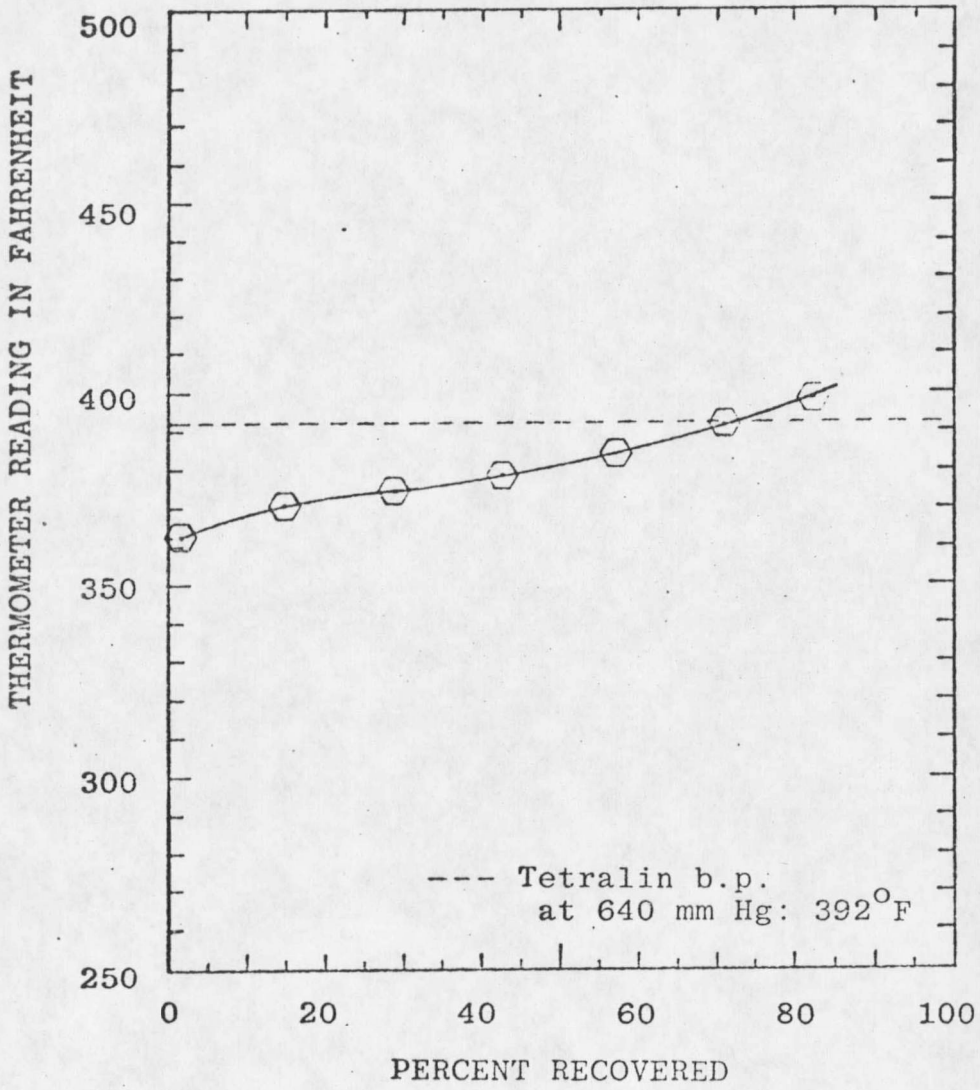


FIGURE 36. DISTILLATION DATA OF RUN 25
COMPOSITE PRODUCT

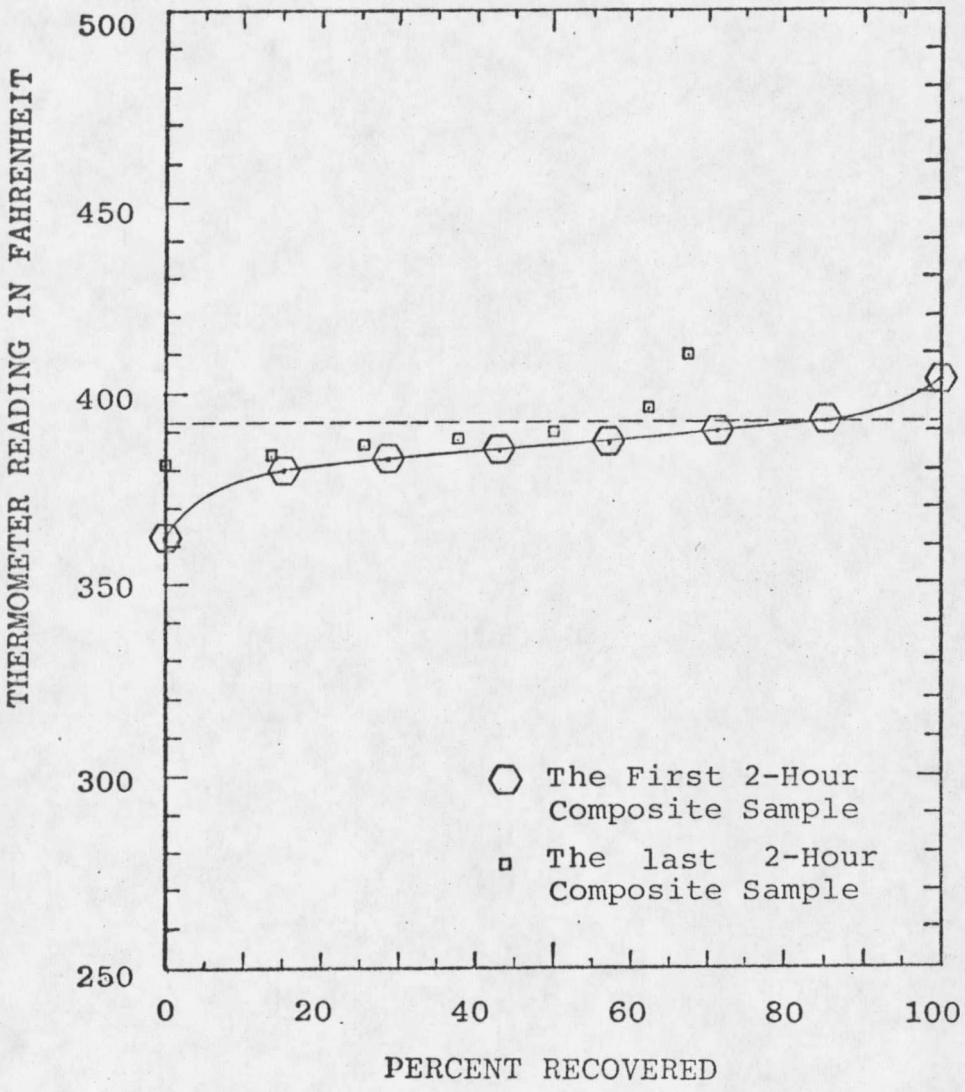


FIGURE 37. DISTILLATION DATA OF RUN 30 WITH MSU KN C5 CATALYST. COMPOSITE SAMPLES OF THE FIRST AND LAST TWO HOURS OF 16-HOUR OPERATION.

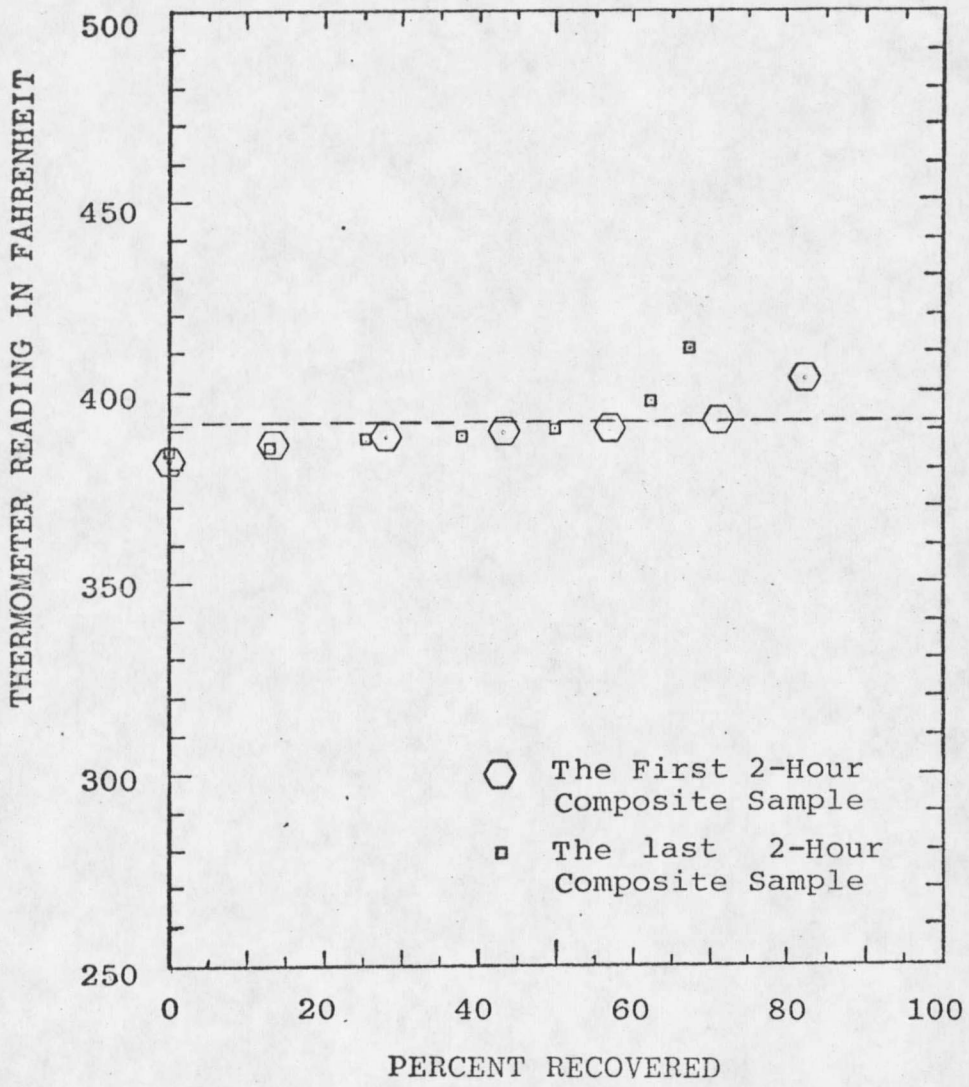


FIGURE 38. DISTILLATION DATA OF RUN 30R1 WITH THE FIRST-REGENERATED MSU KN C5 CATALYST. COMPOSITE SAMPLES OF THE FIRST AND LAST TWO HOURS OF 14-HOUR OPERATION

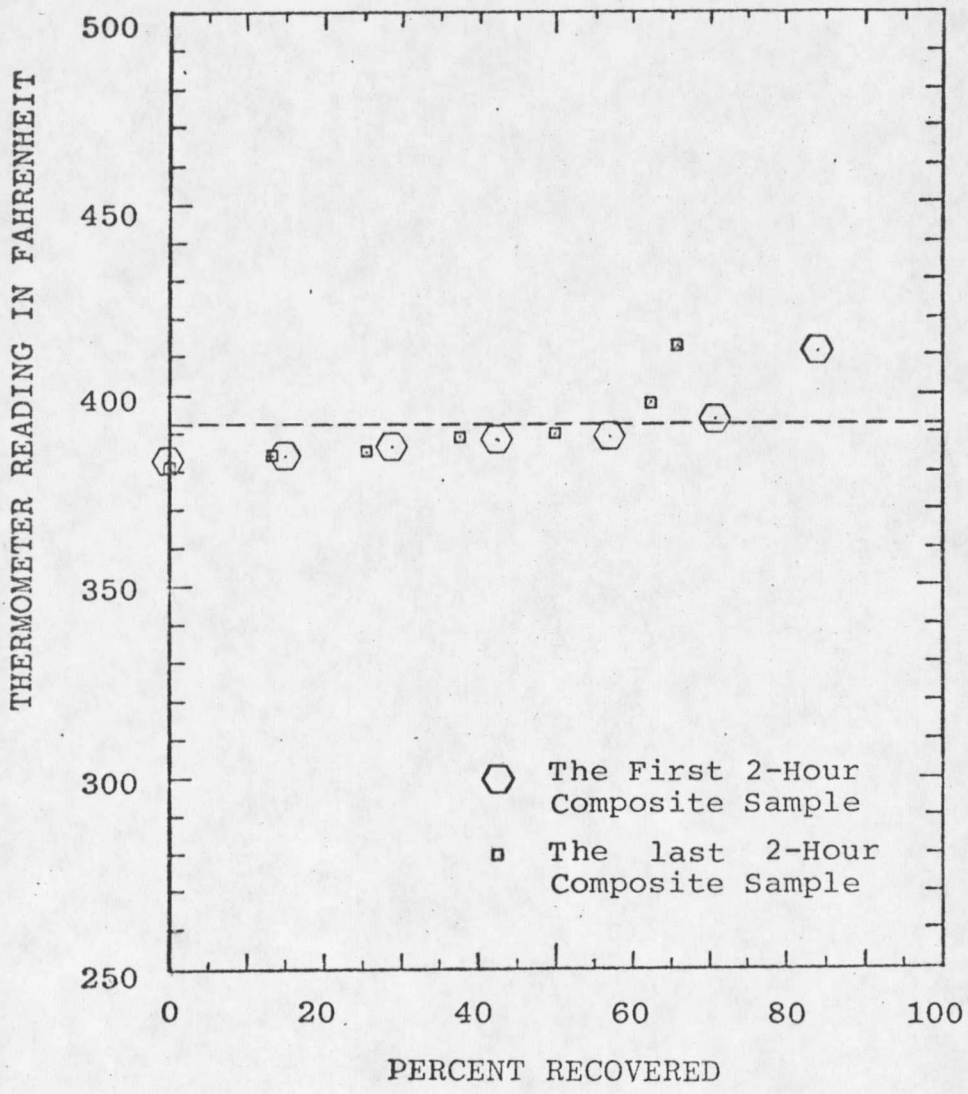


FIGURE 39. DISTILLATION DATA OF RUN 30R2 WITH THE SECOND-REGENERATED MSU KN C5 CATALYST. COMPOSITE SAMPLES OF THE FIRST AND LAST TWO HOURS OF 15-HOUR OPERATION.

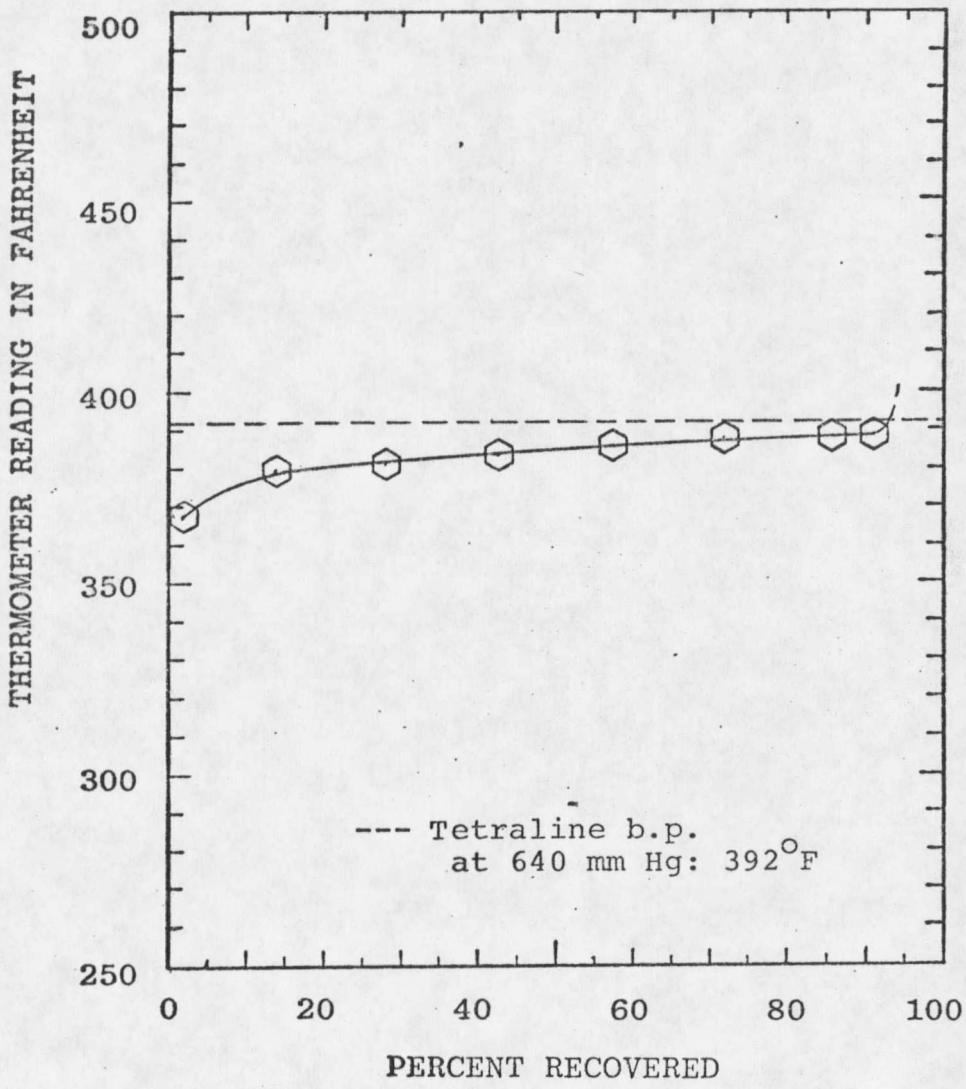


FIGURE 40. DISTILLATION DATA OF RUN 31 COMPOSITE PRODUCT.

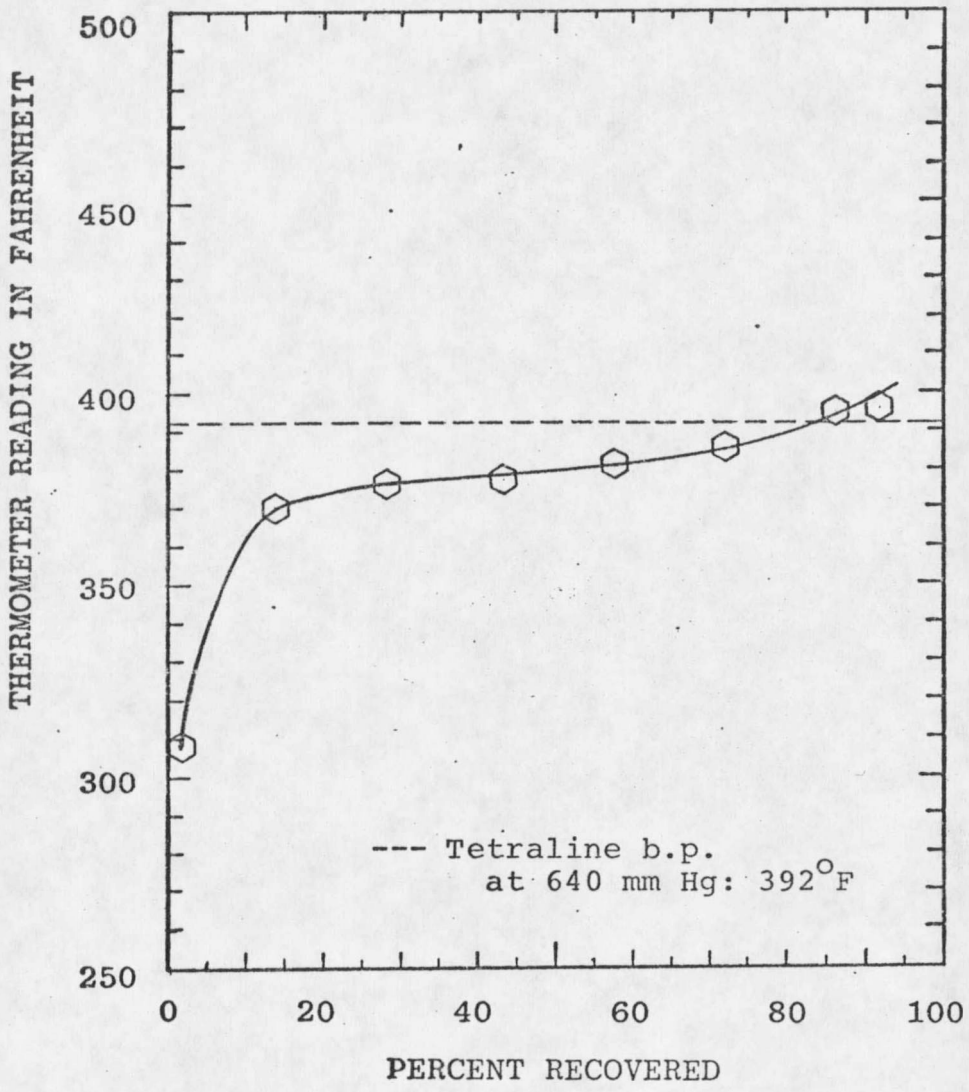


FIGURE 41. DISTILLATION DATA OF RUN 32 COMPOSITE PRODUCT.

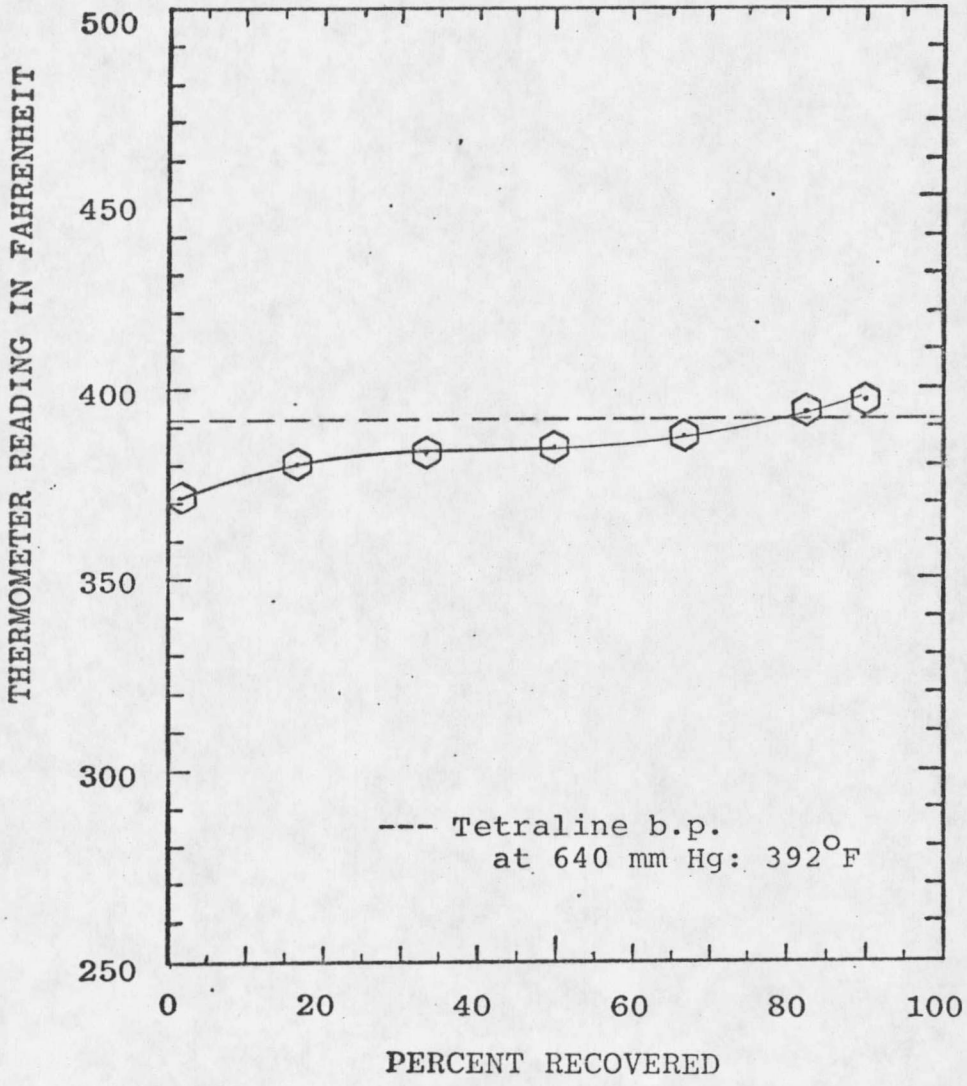


FIGURE 42. DISTILLATION DATA OF RUN 33 COMPOSITE PRODUCT.

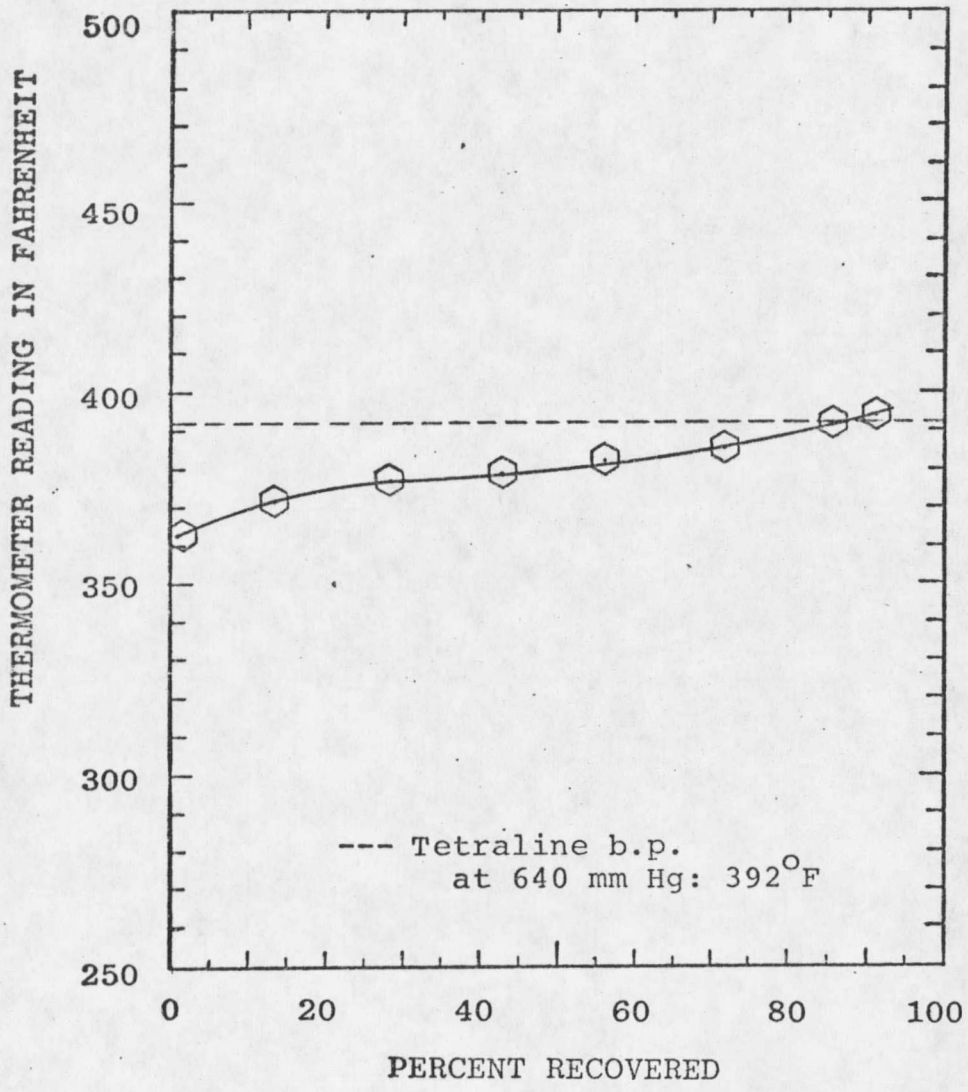


FIGURE 43. DISTILLATION DATA OF RUN 34 COMPOSITE PRODUCT.

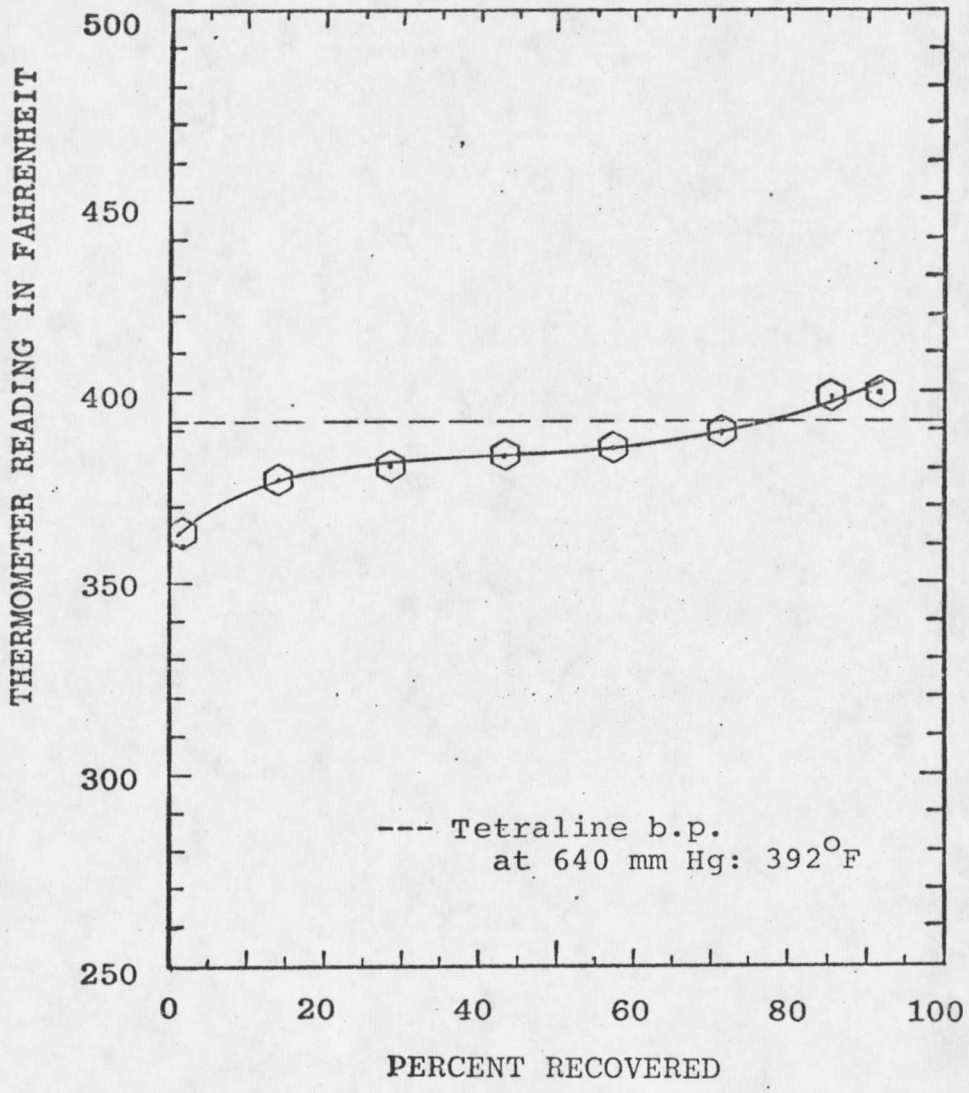


FIGURE 44. DISTILLATION DATA OF RUN 35 COMPOSITE PRODUCT.

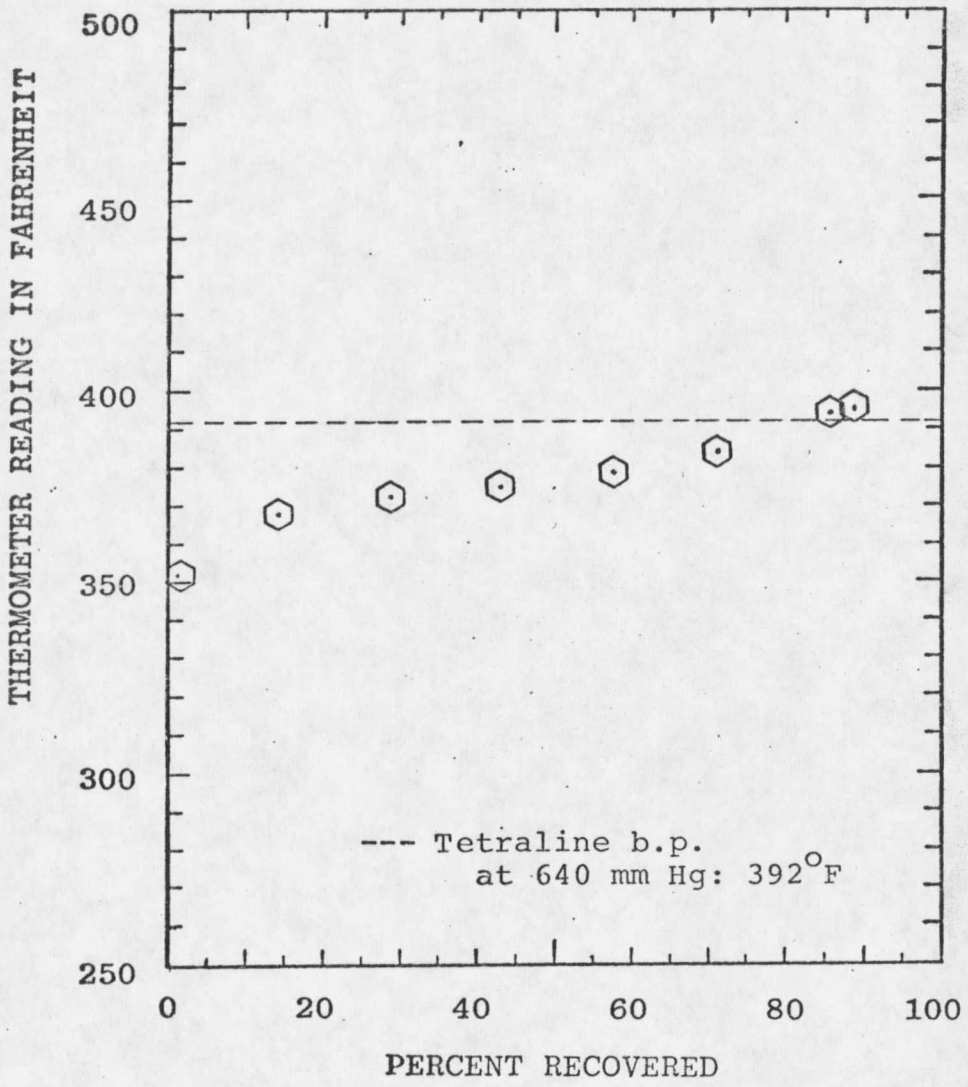


FIGURE 45. DISTILLATION DATA OF RUN 36 COMPOSITE PRODUCT.

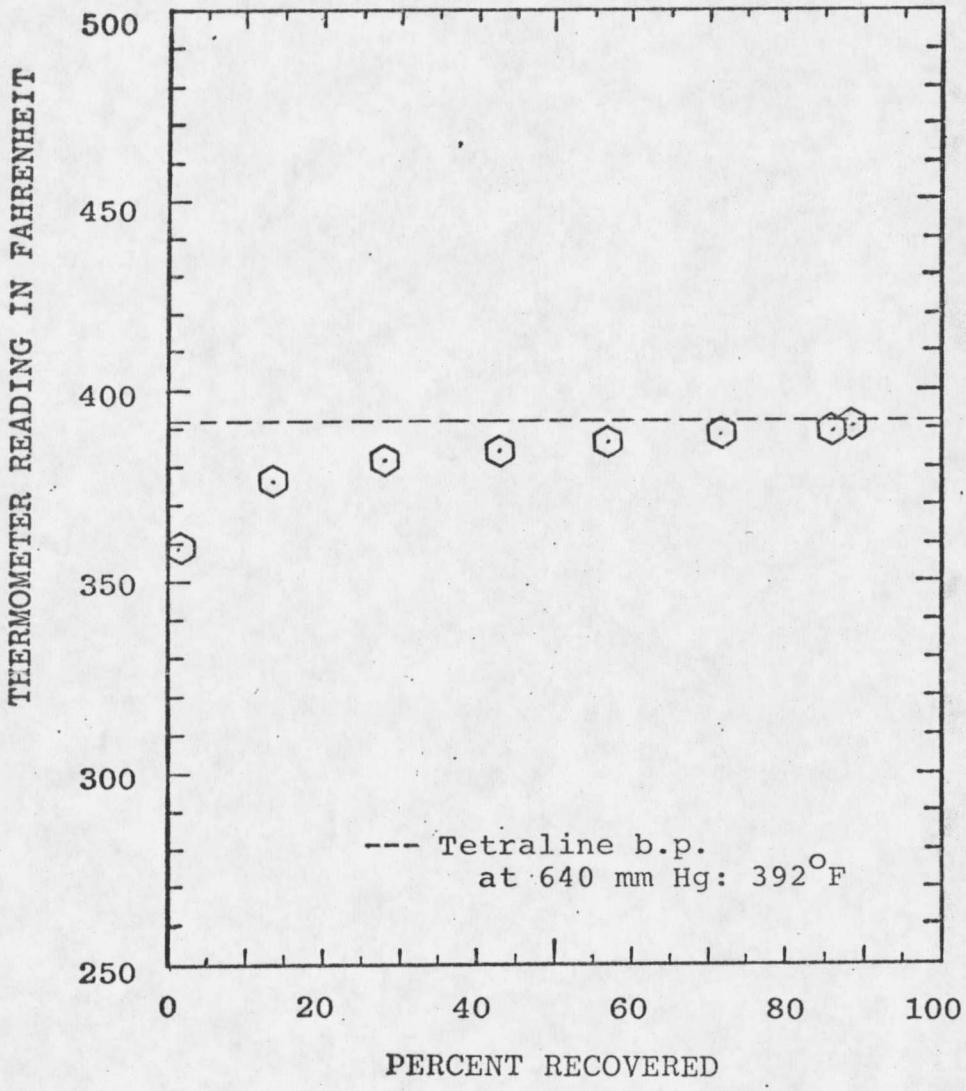


FIGURE 46. DISTILLATION DATA OF RUN 37 COMPOSITE PRODUCT.

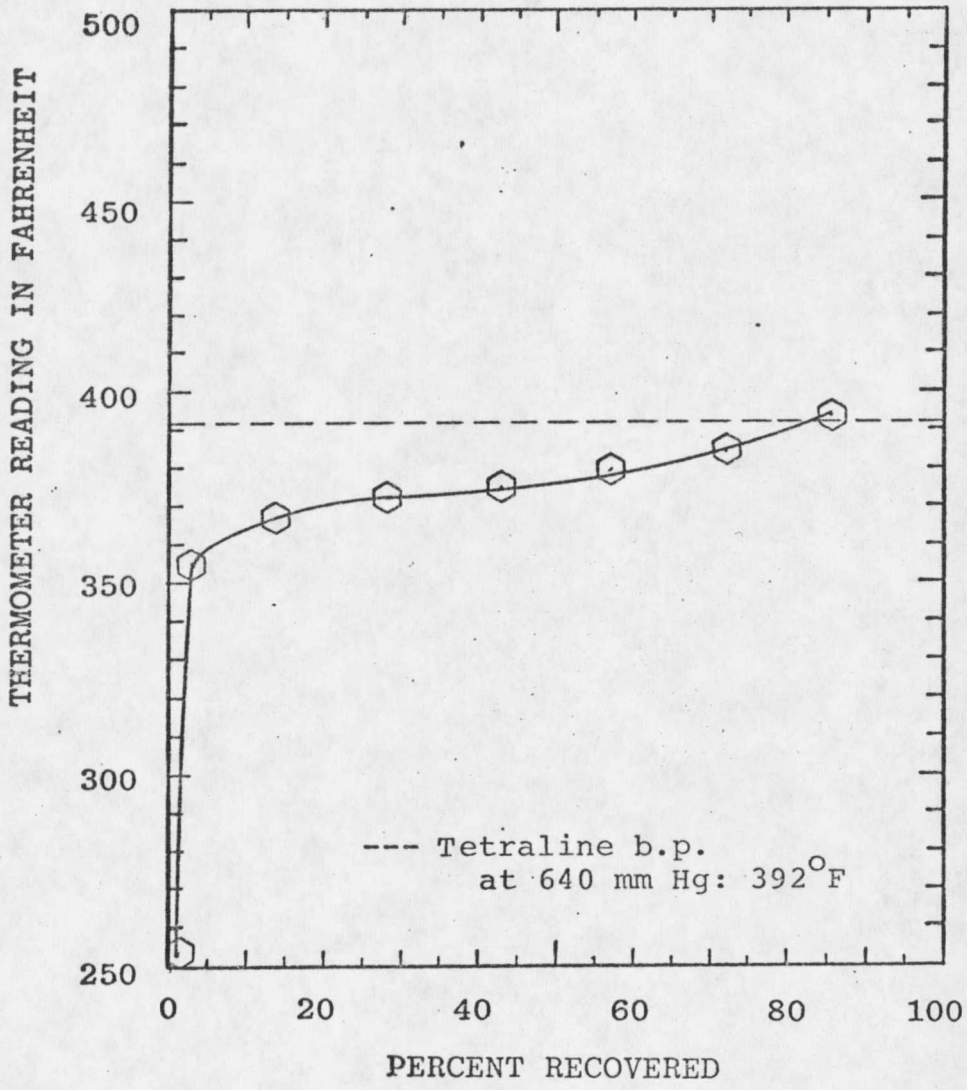


FIGURE 47. DISTILLATION DATA OF RUN 38 COMPOSITE PRODUCT.

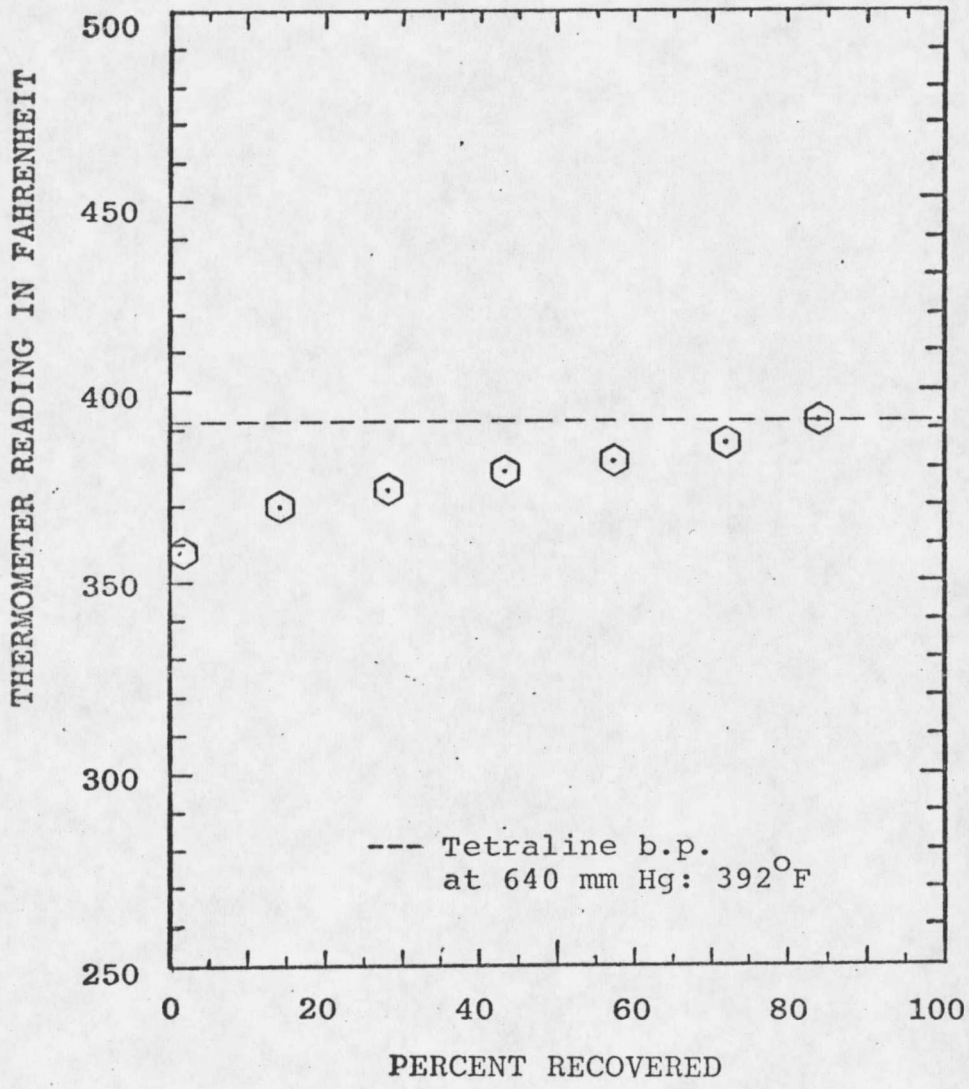


FIGURE 48. DISTILLATION DATA OF RUN 39
COMPOSITE PRODUCT.

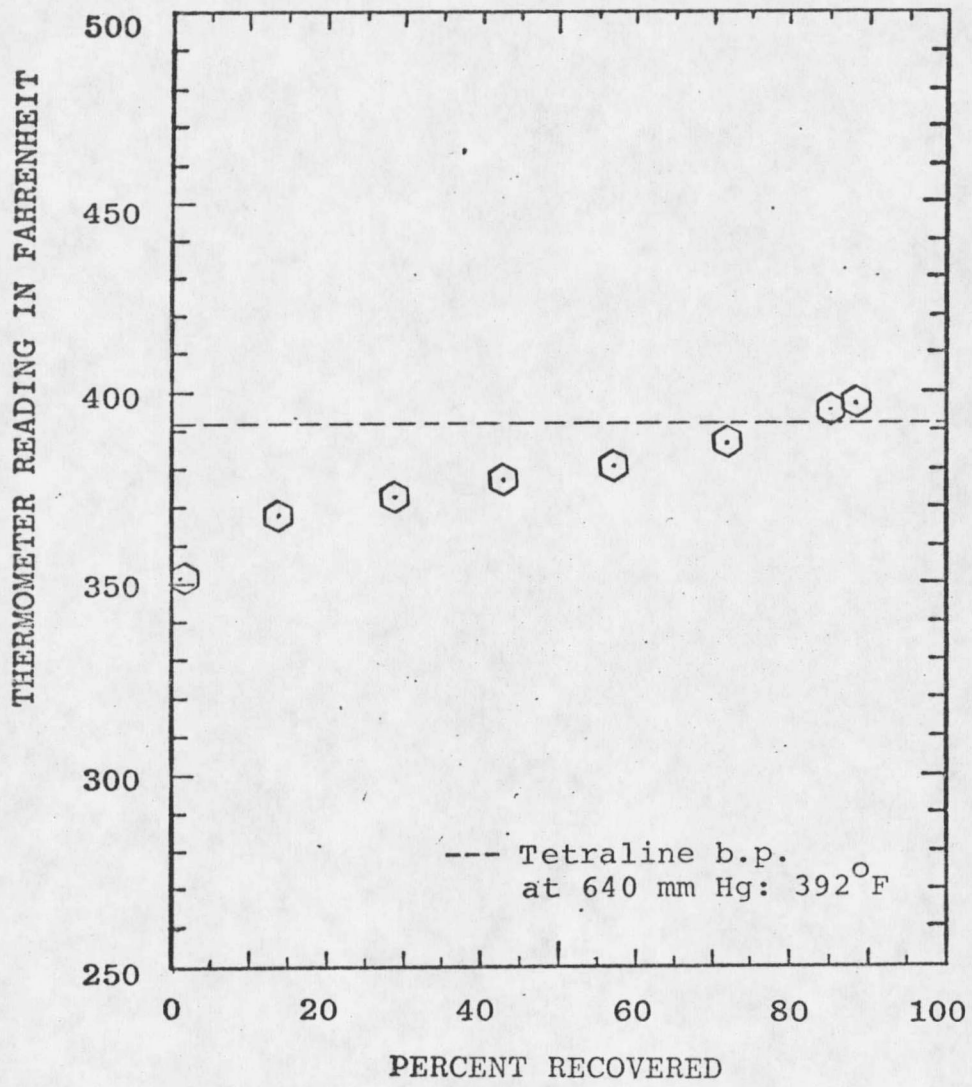


FIGURE 49. DISTILLATION DATA OF RUN 40 COMPOSITE PRODUCT.

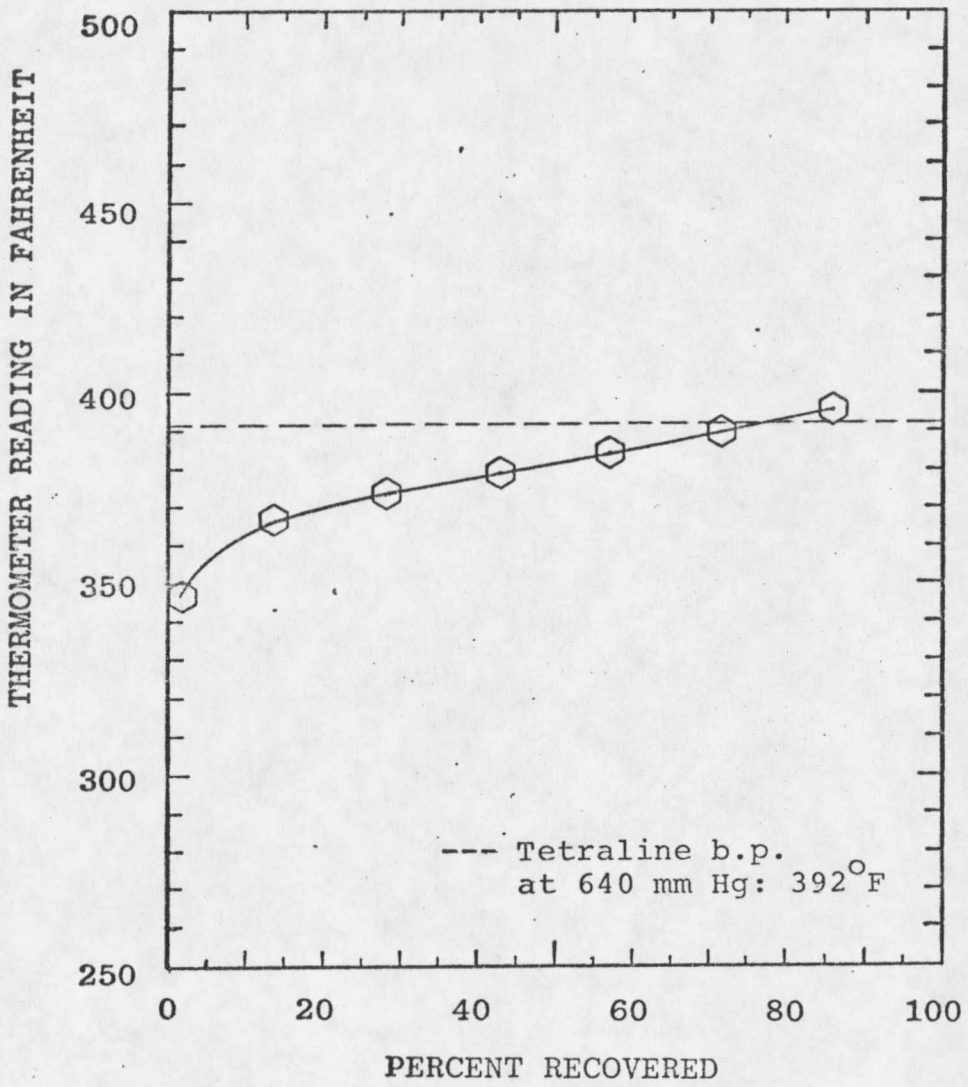


FIGURE 50. DISTILLATION DATA OF RUN 41
COMPOSITE PRODUCT.

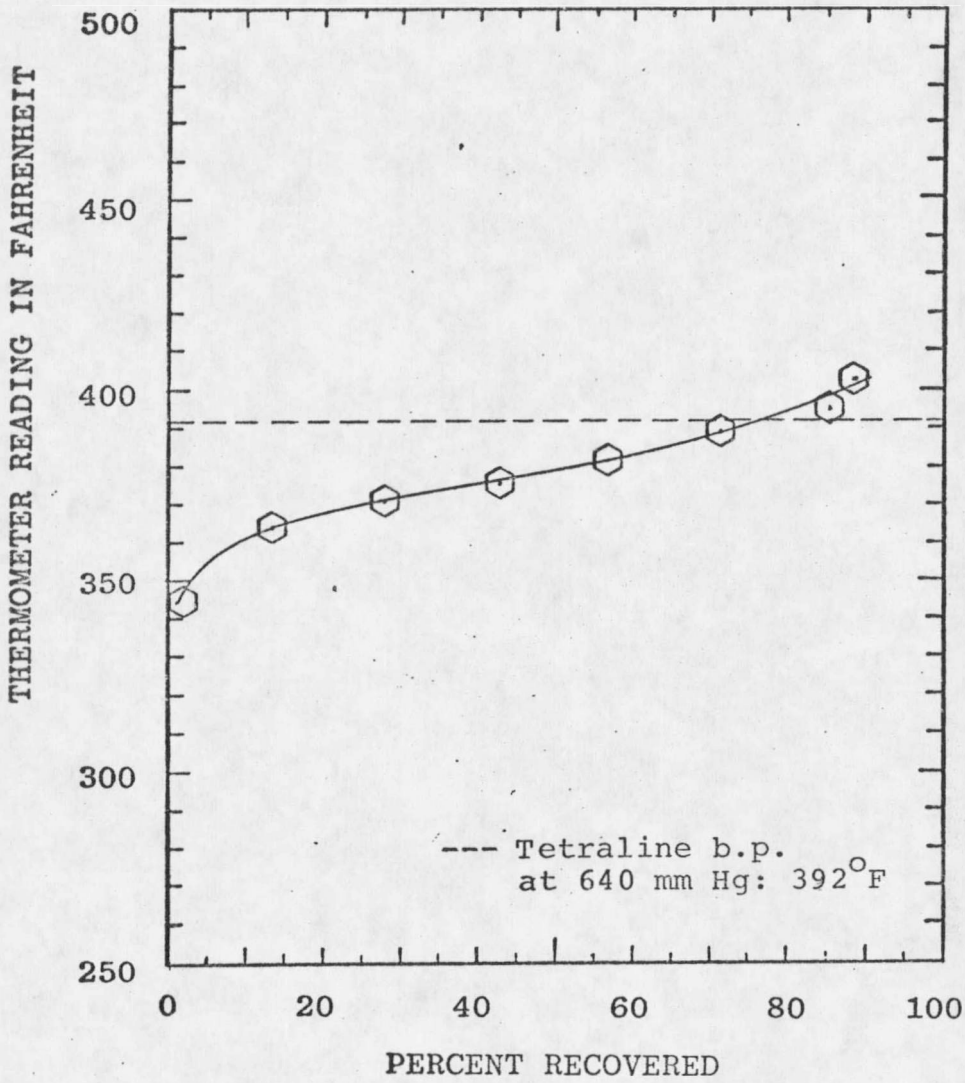


FIGURE 51. DISTILLATION DATA OF RUN 42
COMPOSITE PRODUCT.

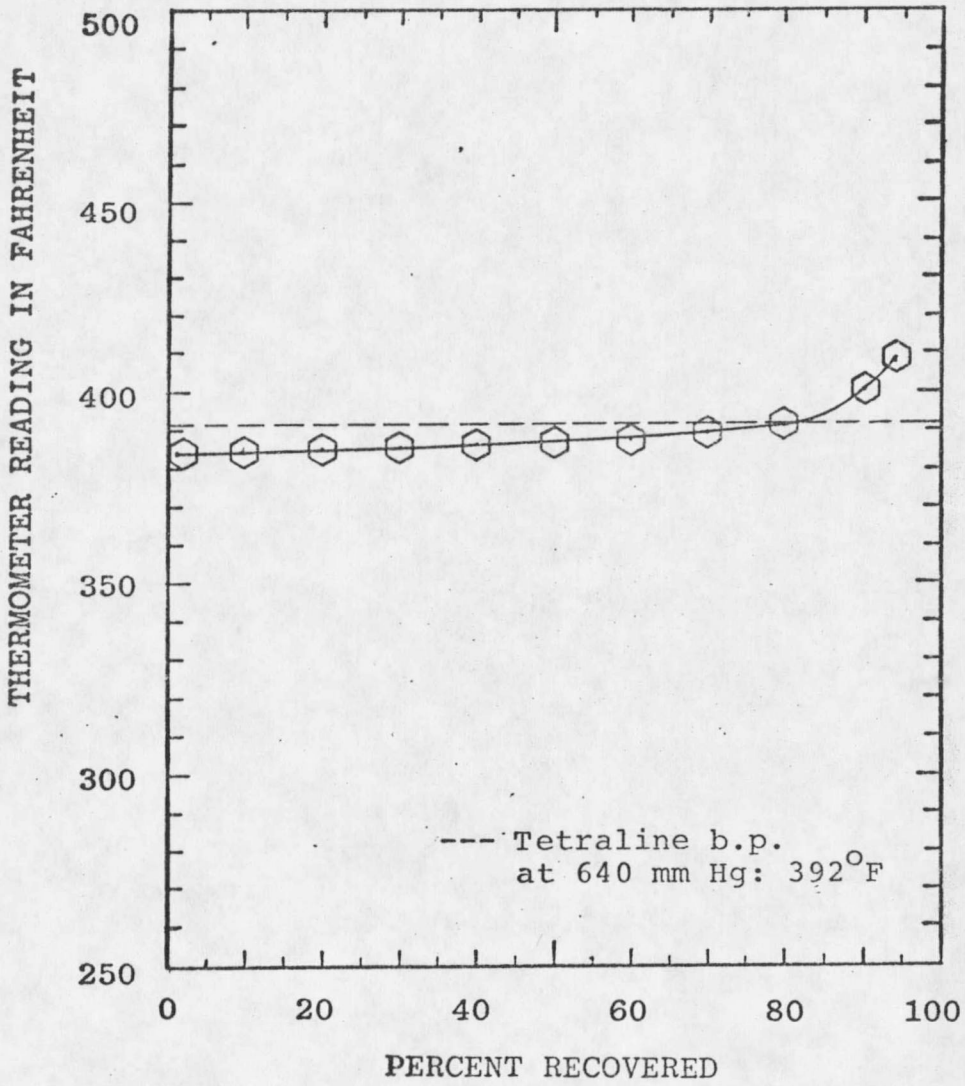


FIGURE 52. DISTILLATION DATA OF TETRALINE USED IN THIS RESEARCH.

APPENDIX B

DATA SHEET FOR RUNS

Run No.: 1

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:1

Catalyst No.: MSU C30D (A. Yeh's) Base: Nalco D
Metal, wt%: MoO_3= 10.7 CoO = 3.06
NiO = 7.16 WO_3 = 8.48

Run Condition
Temp., C : 425 10 LHSV : 1.0 0.2
Press., psig: 1050 - 2100 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 57.0

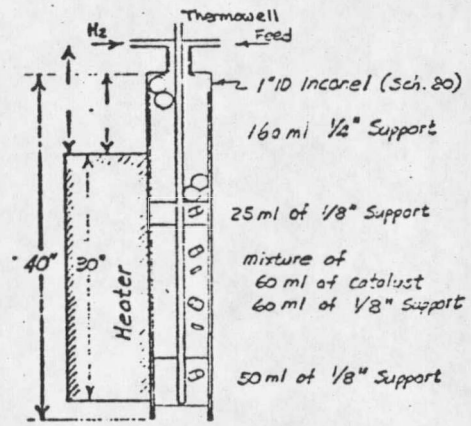
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.845	0.32	135	-	-
30	0.0	0.166	150	0.066	0.25
45	0.0	0.23	165		
60	0.0	0.21	180		
75	0.0	0.15			
90	0.0	0.155			
105	0.0	0.155			
120	0.0	0.18			

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 2

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:1

Catalyst No.: MSU C33B (A. Yeh's) Base: Nalco B
Metal, wt%: MoO_3= 12.4 CoO = 7.07
NiO = 2.7 WO_3 = 10.08

Run Condition
Temp., C : 440 LHSV : 1.0 0.1
Press., psig: 1100 - 1300 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 47.0

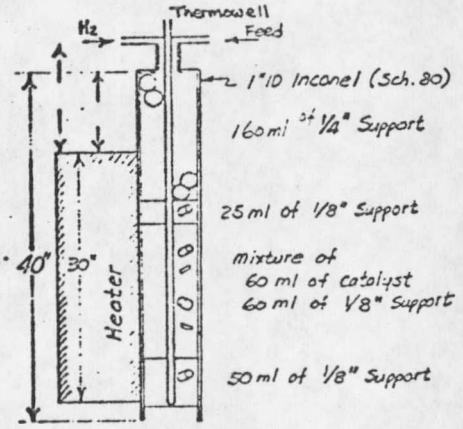
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.375	0.326	135		
30	0.015	0.225	150		
45	0.065	0.23	165		
60	0.045	0.32	180		
75	0.155	0.26	195		
90	0.065		210		
105			225		
120			240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 3

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:1.5

Catalyst No.: MSU C33B Base: Nalco B
Metal, wt%: MoO_3 = 12.4 CoO = 7.07
NiO = 2.7 WO_3 = 10.08

Run Condition
Temp., C : 425 (?) LHSV : 1.0
Press., psig: 1070 - 1300 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 66.0

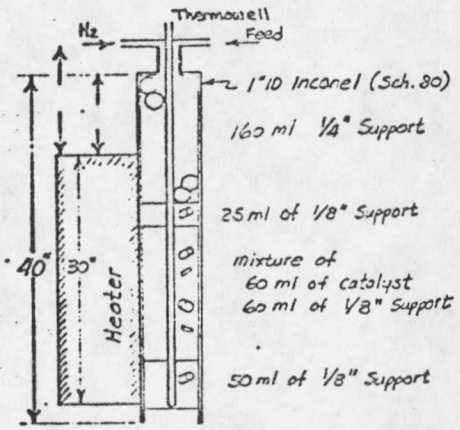
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.375	0.326	135		
30	0.365		150	0.29	
45	0.24		165		
60	0.225	0.388	180	0.35	
75	0.335	0.27	195		
90			210		
105	0.33		225	0.38	0.375
120	0.34		240	0.375	

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 3 continue

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:1.5

Catalyst No.: MSU C33B Base: Nalco B
Metal, wt%: MoO_3= 12.4 CoO = 7.07
NiO = 2.7 WO_3 = 10.08

Run Condition
Temp., C : 425 (?) LHSV : 1.0
Press., psig: 1070 - 1300 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 66.0

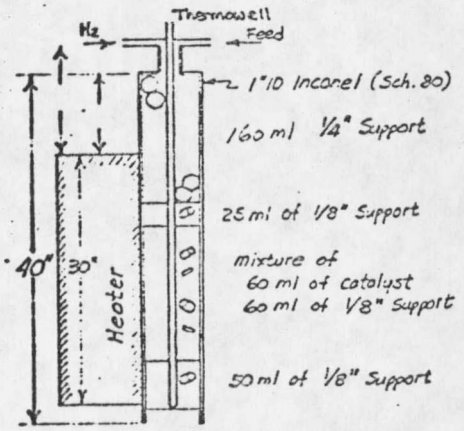
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
255	0.29		375		
270	0.32		390	0.34	
285			405		
300			420		
315	0.30		435		
330			450		
345			465		
360			480		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 4

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:1.5

Catalyst No.: MSU C33B (1st Regenera- Base: Nalco B
Metal, wt%: MoO_3 = tion) CoO =
NiO = WO_3 =

Run Condition
Temp., C : 425 5
Press., psig: 1000
LHSV : 1.0
H_2:Feed; 10,000 scf/bbl

Yield of Oil, Vol.%: 65.7

Time vs. Nitrogen-Sulfur Content

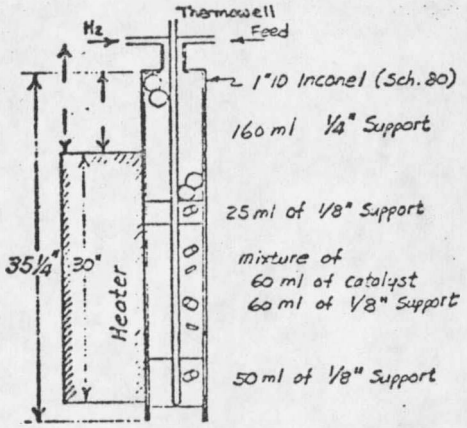
Min.	N	S	Min.	N	S
Feed	0.585	0.29	135	0.40	
30	0.165	0.105	150	0.445	
45			165	0.485	
60	0.15	0.33	180	0.441	0.275
75			195		
90	0.405	0.184	210		
105	0.36	0.215	225		
120	0.371		240		

ASTM Distillation
Volume of Charge, ml: 50

Vol.	IBP (220)
5	365
10	381
15	384
20	384
25	386
30	387
35	388
40	394
42.8	419
50	

Final Volume, ml: 43

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 5

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:2

Catalyst No. MSU C33B (2nd Regenerat- Base: Nalco B
Metal, wt%: MoO_3= ion) CoO =
NiO = WO_3 =

Run Condition
Temp., C : 425 5
Press., psig: 1000
LHSV : 1.0 0.1
H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 78.1

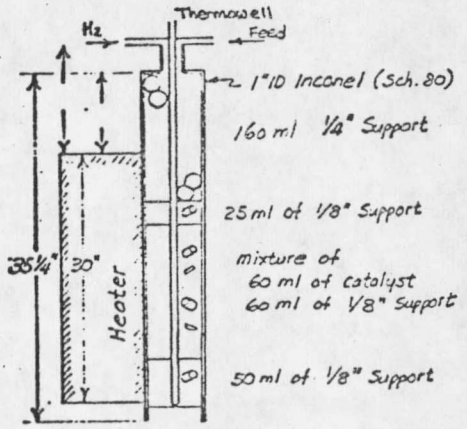
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.604	0.348	135		
30	0.05	0.337	150		
45	0.13		165		
60	0.11		180		
75	0.322		195		
90	0.326		210		
105	0.346		225		
120		0.22	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 6

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSUKND1 Base: Nalco D
Metal, wt%: MoO_3 = 6.54 CoO = 7.06
NiO = 2.22 WO_3 = 9.48

Run Condition
Temp., C : 420 to 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 71.0%

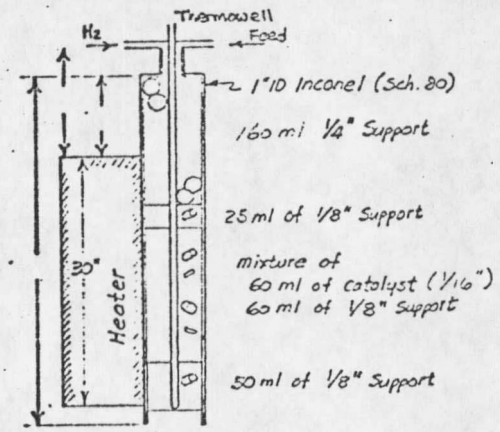
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.42	0.28	135		
30	0.235		150		
45	0.19		165		
60	0.29	0.17	180		
75			195		
90	0.20		210		
105	0.15		225		
120	0.23	0.095	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 7

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSUKN A1 Base: Nalco A
Metal, wt%: MoO_3 = 8.42 CoO = 4.00
NiO = 1.29 WO_3 = 10.04

Run Condition
Temp., C : 425°C LHSV : 1.0 + 0.1
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 76.4

Time vs. Nitrogen-Sulfur Content

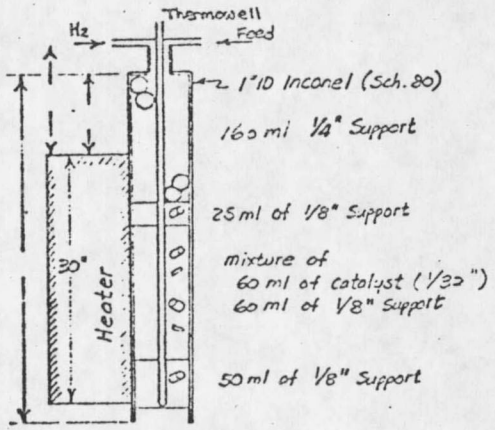
Min.	N	S	Min.	N	S
Feed	0.47	0.275	135		
30	0.25	0.095	150	0.26	0.065
45	0.315	0.05	165		
60	0.21		180	0.23	
75	0.335		195		
90	0.30	0.095	210	0.195	0.07
105	0.32		225		
120	0.21		240		

ASTM Distillation
Volume of Charge, ml: 51.5

Vol.	IBP (142°C)	or 288°F
5	183	361
10	196	385
15	201	394
20	202	396
25	203	397
30	204	399
35	205	401
40	210	410
45	312	594
50		

Final Vol.,ml: 45.2

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 8

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN B1 Base: Nalco B
Metal, wt%: MoO_3 = 8.91 CoO = 4.28
NiO = 2.28 WO_3 = 9.93

Run Condition
Temp., C : 420°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 71.3

Time vs. Nitrogen-Sulfur Content

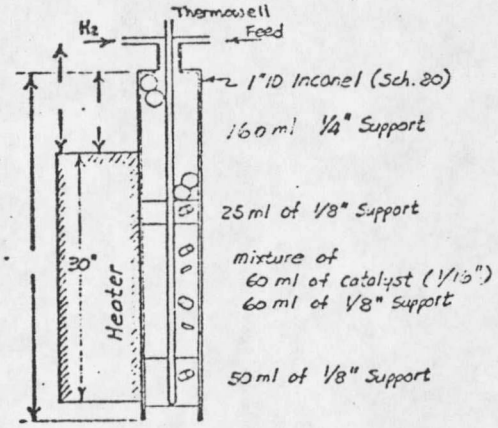
Min.	N	S	Min.	N	S
Feed	0.51	0.27	135		
30	0.17	0.10	150		
45	0.23	0.105	165		
60	0.20		180		
75	0.20	0.085	195		
90	0.19	0.045	210		
105	0.20		225		
120	0.20	0.053	240		

ASTM Distillation
Volume of Charge, ml: 50.0

Vol.	IBP (321°F)
5	342
10	368
15	372
20	376
25	380
30	381
35	383
40	390
45	408
46.7	411

Final Vol., ml: 47.0

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 9

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:2

Catalyst No.: MSU KN E1 Base: Nalco E
Metal, wt%: MoO_3= 6.09 CoO = 4.0
NiO = 2.26 WO_3 = 10.32

Run Condition
Temp., C : 425 to 450°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 89.17

Time vs. Nitrogen-Sulfur Content

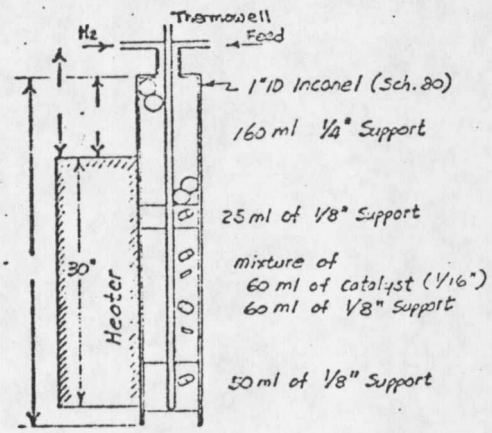
Min.	N	S	Min.	N	S
Feed	0.43	0.275	135		
30	0.145	0.22	150		
45	0.20	0.065	165		
60	0.18		180		
75	0.15		195		
90	0.20	0.07	210		
105	0.22		225		
120	0.16	0.08	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (255°F)
5	365
10	379
15	388
20	390
25	392
30	394
35	396
40	399
45	410
47.5	482

Final Vol., ml: 48.1

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 10

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN A2 Base: Nalco A
Metal, wt%: MoO_3 = 11.00 CoO = 4.84
NiO = 2.70 WO_3 = 10.20

Run Condition
Temp., C : 425° LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 68.7

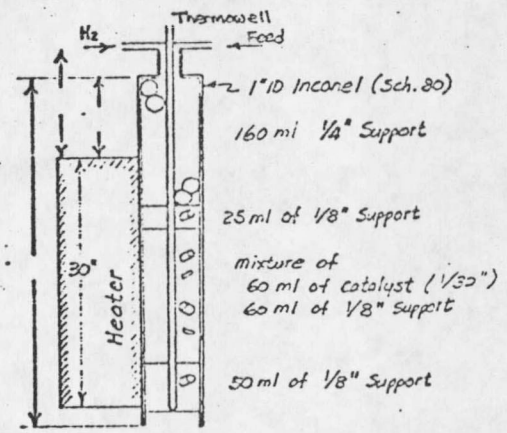
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.49		135		
30	0.26		150	0.045	
45	0.085		165		
60	0.105		180		
75	0.085		195		
90	0.06		210		
105	0.11		225		
120	0.075		240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 11

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: Blank Base: Nalco C
Metal, wt%: MoO_3 = CoO =
NiO = WO_3 =

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 45.0

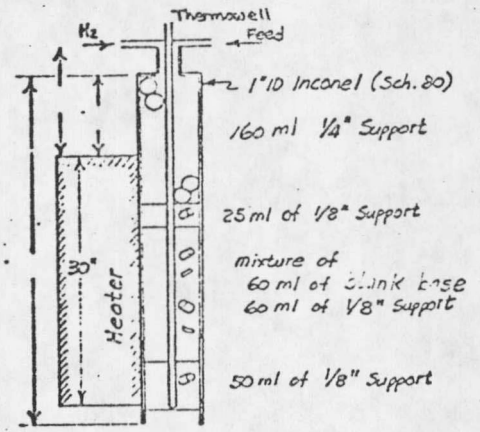
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.60		135		
30	0.29		150		
45			165		
60	0.07		180		
75			195		
90			210		
105			225		
120			240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 12

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: Blank Base: Nalco C
Metal, wt%: MoO_3 = ____ CoO = ____
NiO = ____ WO_3 = ____

Run Condition
Temp., C : 425° LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 59.17

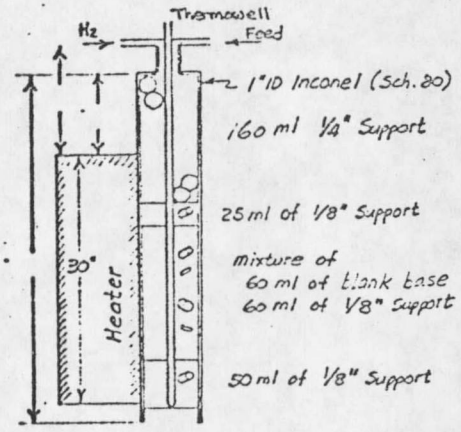
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.58		135		
30	0.265		150		
45			165		
60	0.05		180		
75			195		
90	0.29		210		
105			225		
120	0.285		240		

ASTM Distillation
Volume of Charge, ml: ____

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 13

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN D2 Base: Nalco D
Metal, wt%: MoO_3 = 11.48 CoO = 5.10
NiO = 2.84 WO_3 = 10.19

Run Condition
Temp., C : 425° to 430°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 57.08

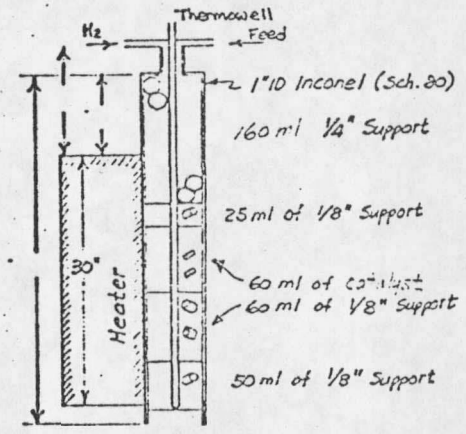
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.34		135		
30			150		
45	0.065		165		
60	0.065		180		
75	0.255		195		
90	0.235		210		
105	0.26		225		
120	0.255		240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 14

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: Blank Base: Nalco E
Metal, wt%: MoO_3 = ____ CoO = ____
NiO = ____ WO_3 = ____

Run Condition
Temp., C : 425° LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 64.17

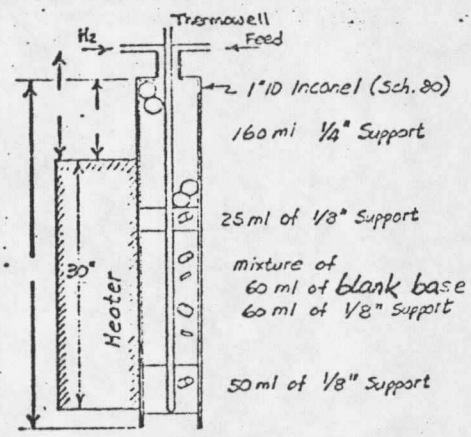
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.50		135		
30	0.045		150		
45	0.07		165		
60	0.24		180		
75			195		
90	0.33		210		
105			225		
120	0.35		240		

ASTM Distillation
Volume of Charge, ml: ____

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 15

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: Blank Base: Nalco C
Metal, wt%: MoO_3 = CoO =
NiO = WO_3 =

Run Condition
Temp., C : 410 LHSV : 0.18
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 57

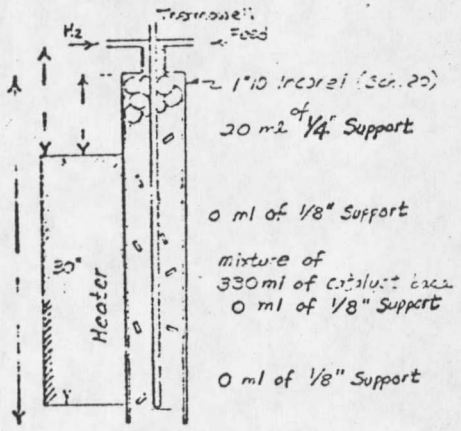
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.315	0.27	135		
30	0.075	0.088	150	0.005	
45	0.073		165		
60	0.072	0.086	180		
75	0.073		195		
90	0.086	0.08	210		
105	0.035		225		
120	0.027	0.081	240		

ASTM Distillation
Volume of Charge, ml: 31

Vol.	IBP (380 °F)
5	382
10	384
15	385
20	386
25	388
29	404
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.6397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 16

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: Blank Base: Norton ¼"
Metal, wt%: MoO_3 = CoO = Denstone
NiO = WO_3 =

Run Condition
Temp., C : 425 LHSV : 0.17
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 80.8

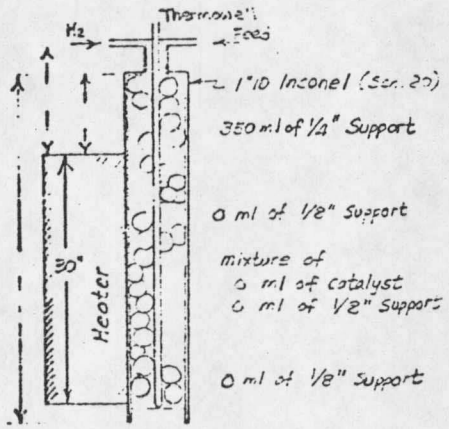
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.315	0.27	135		
30	0.13	0.31	150		
45			165		
60	0.145		180		
75	0.255	0.245	195		
90	0.		210		
105	0.27		225		
120	0.275		240		

ASTM Distillation
Volume of Charge, ml: 26

Vol.	IBP (380 °F)
5	384
10	385
15	387
20	402
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 17

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN B2 Base: Nalco B
Metal, wt%: MoO_3 = 11.1 CoO = 4.86
NiO = 2.62 WO_3 = 10.47

Run Condition
Temp., C : 440 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10000 scf/bbl

Yield of Oil, Vol.%: 67.5

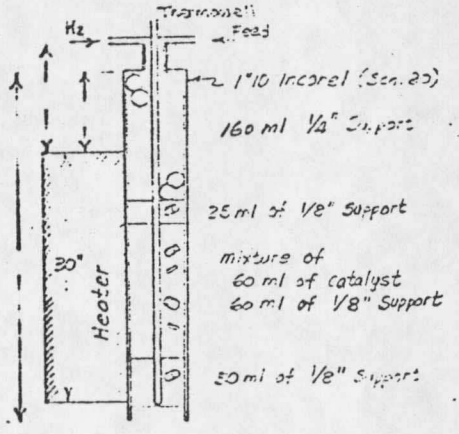
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.22	0.25	135		
30	0.091	0.17	150		
45	0.075		165		
60	0.05	0.225	180		
75	0.04		195		
90	0.04	0.195	210		
105	0.045		225		
120	0.04	0.22	240		

ASTM Distillation
Volume of Charge, ml: 26

Vol.	IBP (368 °F)
5	376
10	380
15	384
20	390
24.5	402
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 18

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN E2 Base: Nalco E
Metal, wt%: MoO_3 = 10.18 CoO = 4.88
NiO = 2.84 WO_3 = 10.68

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 70.42

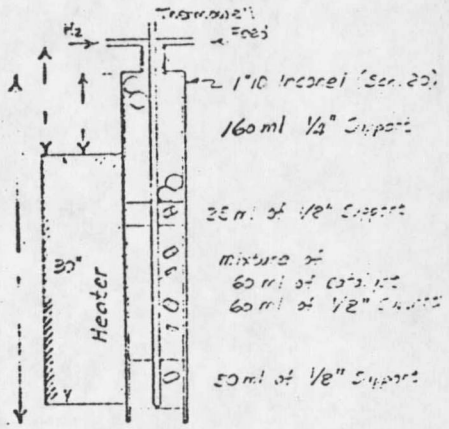
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.22	0.25	135		
30	0.075	0.15	150		
45	0.05		165		
60	0.065	0.179	180		
75	0.07		195		
90	0.085	0.13	210		
105	0.075		225		
120	0.07		240		

ASTM Distillation
Volume of Charge, ml: 31

Vol.	IBP (358 °F)
5	368
10	372
15	375
20	380
25	391
28	403
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 19

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C1 Base: Nalco C
Metal, wt%: MoO_3 = 8.32 (.0908) CoO = 0.00
NiO = 0.00 WO_3 = 0.00
( ) metal in gm per gm base

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 63.8

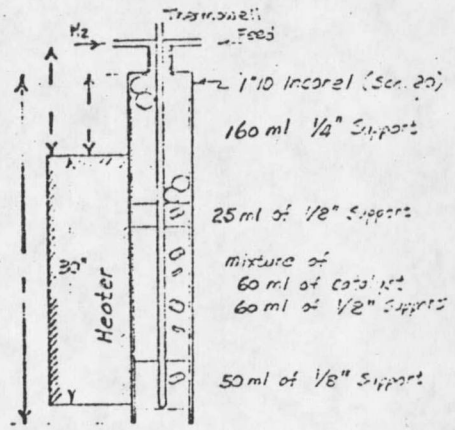
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.255	0.25	135		
30	0.033		150		
45	0.035		165		
60	0.033	0.175	180		
75	0.04		195		
90	0.033	0.66 (?)	210		
105	0.039		225		
120	0.029	0.52 (?)	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 20

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C2 Base: Nalco C
Metal, wt%: MoO_3=7.99 (.0908) CoO = 0.00
NiO =3.98 (.0452) WO_3 = 0.00
( ) metal in gram per i gram base

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 76.3

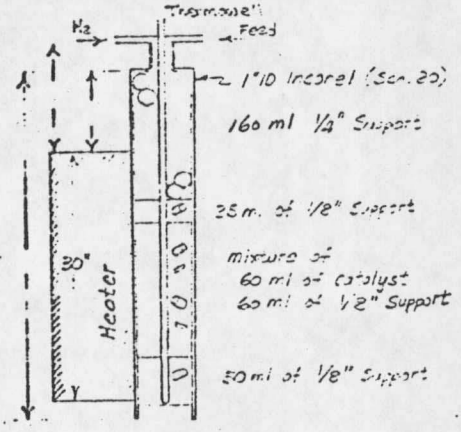
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.22	0.26	135		
30	0.026		150	0.058	
45	0.031		165		
60	0.044		180		
75	0.03		195		
90	0.023	0.094	210		
105	0.039		225		
120	0.020	0.08	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 21

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C3 Base: Nalco C
Metal, wt%: MoO_3=7.4 (.0908) CoO = 7.42 (.0911)
NiO =3.69(.0453) WO_3 = 0.00
( ) metal in gram per 1 gram base

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 67.1

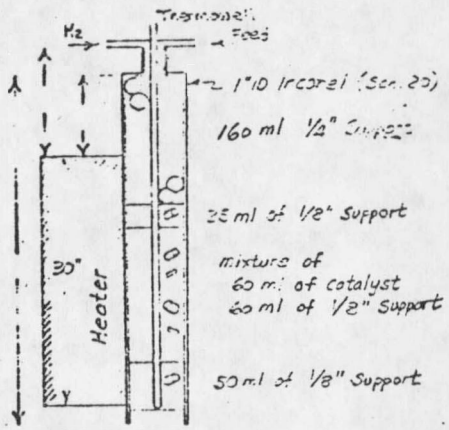
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.265	0.275	135		
30	0.049		150		
45	0.057		165		
60	0.040		180		
75	0.037		195		
90	0.050	0.12	210		
105	0.059		225		
120	0.05	0.072	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 22

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C4 Base: Nalco C
Metal, wt%: MoO_3=6.45(.0908) CoO = 6.46 (0.0911)
NiO =3.22(.0453) WO_3 =12.89 (0.1816)

Run Condition
Temp., C : 420 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 66.7

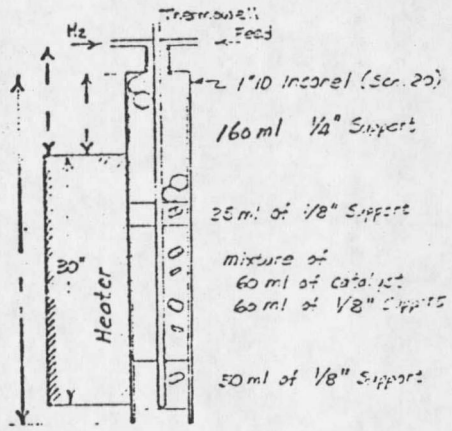
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.255	0.275	135		
30	0.23	0.18	150		
45	0.05		165		
60	0.02		180		
75	0.03		195		
90	0.03		210		
105	0.03		225		
120	0.019	0.29	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 23

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: Mobil TCC Dura Bead Base:
Metal, wt%: MoO_3 = CoO =
NiO = WO_3 =

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 54.2

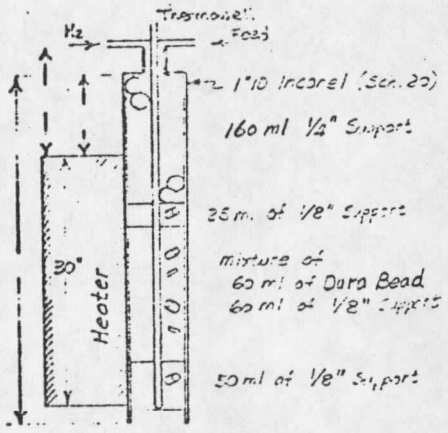
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.255	0.275	135		
30			150		
45	0.078		165		
60	0.03	1.39	180		
75	0.04		195		
90	0.029	0.52	210		
105	0.03		225		
120	0.039	0.31	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 24

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C5 Base: Nalco C
Metal, wt%: MoO_3=12.08(.1374) CoO = 0.00
NiO = 0.00 WO_3 = 0.00

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 47.9

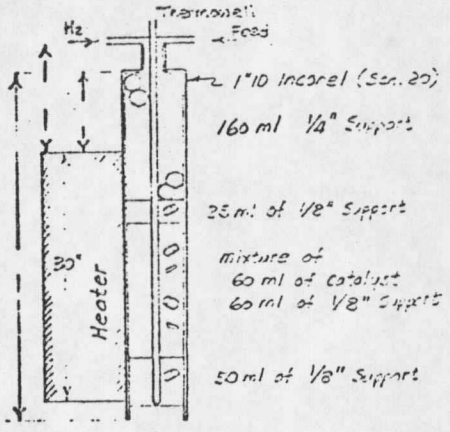
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.30	0.30	135		
30			150		
45	0.00		165		
60	0.00	0.29	180		
75	0.00		195		
90	0.00	0.23	210		
105	0.00		225		
120	0.00	0.14	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 25

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C6 Base: Nalco C
Metal, wt%: MoO_3=11.92(.1374) CoO = 0.00
NiO = 1.34(.0155) WO_3 = 0.00
( ) metal in gram per gram base

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 67.1

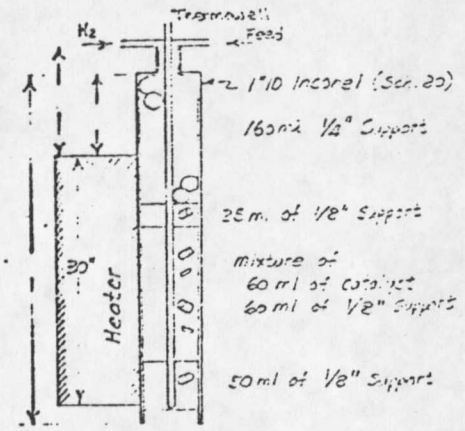
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.30	0.30	135		
30	0.00		150		
45	0.00		165		
60	0.00	0.215	180		
75	0.008		195		
90	0.033	0.071	210		
105	0.00		225		
120	0.017	0.057	240		

ASTM Distillation
Volume of Charge, ml: 35

Vol.	IBP (362 °F)
5	370
10	374
15	378
20	383
25	391
29	398
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 26

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C7 Base: Nalco C
Metal, wt%: MoO_3=11.24(.1374) CoO = 5.74 (.0702)
NiO = 1.27(.0155) WO_3 = 0.00
( ) metal in gram per gram base

Run Condition
Temp., C : 430 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 63.3

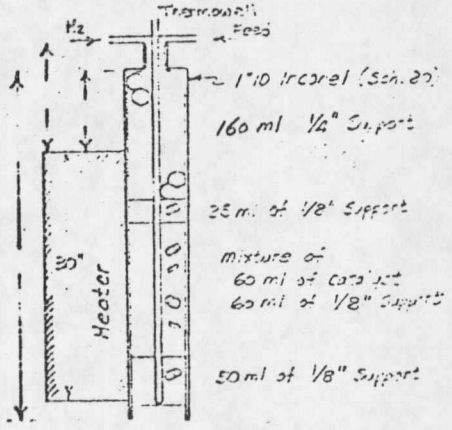
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.27	0.28	135		
30	0.008		150		
45	0.008		165		
60	0.036		180		
75	0.04		195		
90	0.04	0.135	210		
105	0.048		225		
120	0.041		240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 27

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C8 Base: Nalco C
Metal, wt%: MoO_3=10.02(.1374) CoO = 5.12 (.0702)
NiO = 1.13(.0155) WO_3 =10.82 (.1484)
( ) metal in gram per gram base

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 71.2

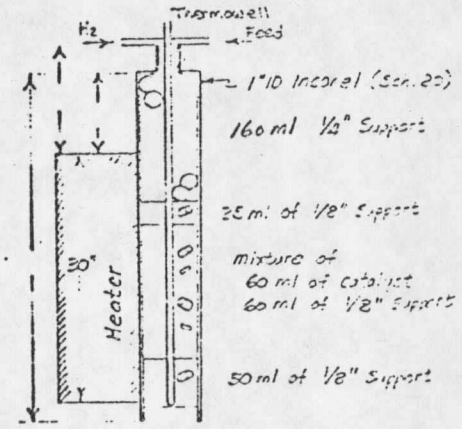
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.27	0.28	135		
30	0.024	0.24	150	0.024	
45	0.02		165		
60	0.016	0.2	180		
75	0.016		195		
90	0.016	0.05	210		
105	0.045		225		
120	0.028	0.039	240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 28

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C9 Base: Nalco C
Metal, wt%: MoO_3 = 4.68(.0491) CoO = 0.00
NiO = 0.00 WO_3 = 0.00

Run Condition
Temp., C : 425 LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 61.7

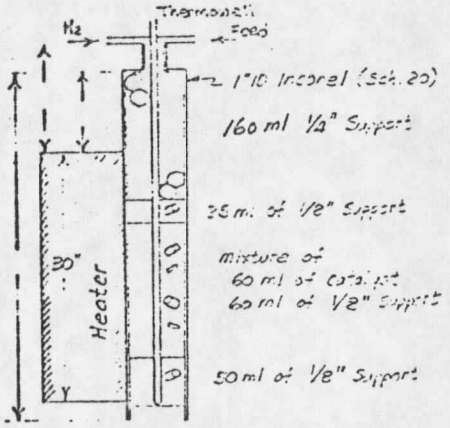
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.27		135		
30	0.036		150		
45	0.033		165		
60	0.04		180		
75	0.036		195		
90	0.036		210		
105	0.036		225		
120	0.032		240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 29

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN 10 Base: Nalco C
Metal, wt%: MoO_3 = 4.62(.049) CoO = 0.00
NiO = 1.32(.014) WO_3 = 0.00
( ) metal oxide in gm per gm base

Run Condition
Temp., C : 425° LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 68.7

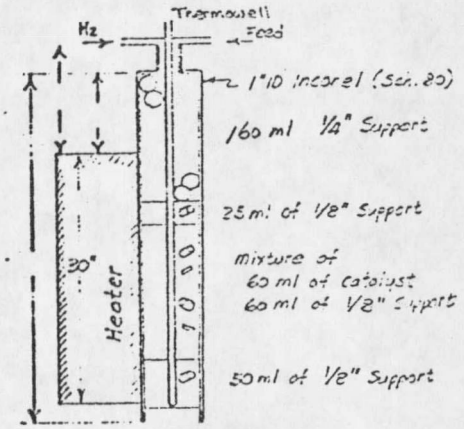
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.24	0.265	135	0.005	
30	0.016		150	0.014	0.075
45	0.024		165	0.014	
60	0.016		180	0.009	0.068
75	0.024		195	0.010	
90	0.008		210	0.011	0.059
105	0.032		225	0.018	
120	0.00	0.155	240	0.025	0.069

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP (°C)
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. Å	Average Pore D. Å *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (Å) = 40,000 (pore volume/surface area)

Run No.: 29 continued

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=_____

Catalyst No.:_____ Base:_____
Metal, wt%: MoO_3 = _____ CoO = _____
NiO = _____ WO_3 = _____

Run Condition
Temp., C : _____ LHSV : _____
Press., psig: _____ H_2:Feed: _____scf/bbl

Yield of Oil, Vol.%:_____

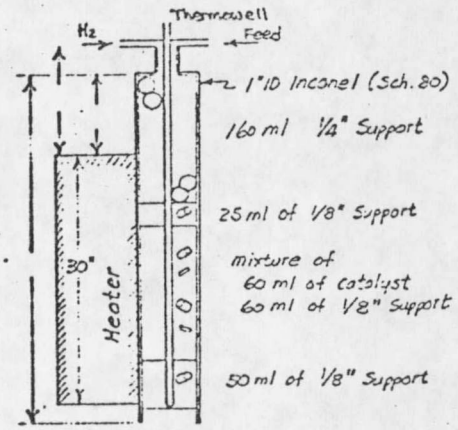
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
255	0.021		375		
270	0.024	0.081	390		
285	0.024		405		
300			420		
315			435		
330			450		
345			465		
360			480		

ASTM Distillation
Volume of Charge, ml: _____

Vol.	IBP
5	
10	
15	
20	
25	
30	
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 30

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C5 Base: Nalco C
Metal, wt%: MoO_3=12.08(.1374) CoO = 0.00
NiO = 0.00 WO_3 = 0.00
( ) metal oxide in gm per gm base

Run Condition
Temp., C : 425° LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 73.9

Time vs. Nitrogen-Sulfur Content

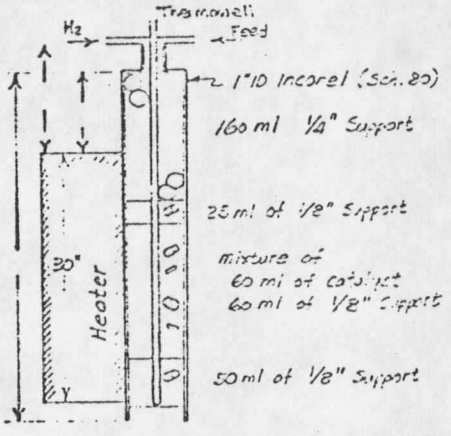
Min.	N	S	Min.	N	S
Feed	See the next page		135		
30			150		
45			165		
60			180		
75			195		
90			210		
105			225		
120			240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP :362 F; 381 F	
5	379	383
10	382	385
15	384	387
20	386	389
25	389	395
30	392	409(27 cc)
35	404	
40		

Charge	35 cc 1st 2hr composite sample	40 cc last 2hr composite sample

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 30 continued

Hour	N	S
Feed	0.225	0.39
(first part of the run)		
0.5	0.016	
0.75	0.004	
1.00	0.00	0.59
1.25	0.00	
1.5	0.016	0.135
1.75	0.004	
2.00	0.00	0.084
2.25	0.012	
2.5	0.00	0.073
2.75	0.00	
3.00	0.00	0.078
3.25	0.00	
3.5	0.00	0.079
3.75	0.04	
4.00	0.008	0.089
4.25	0.00	
4.5	0.00	0.083
4.75	0.008	
5.00	0.024	0.090
5.25	0.013	
5.5	0.032	0.093
5.75	0.049	
6.00	0.028	0.105
Feed	0.21	0.40
(second part of the run)		
6.5	0.04	
6.75	0.041	0.185
7.00	0.033	
7.25	0.024	0.097
7.5	0.024	
7.75	0.020	0.205
8.00	0.028	
8.25	0.040	0.28
8.5	0.049	
8.75	0.041	0.315
9.00	0.032	
9.25	0.056	0.225
9.5	0.040	
9.75	0.065	0.195
10.0	0.065	

Hour	N	S
Feed	0.16	0.415
(third part of the run)		
10.5	0.008	0.084
10.75	0.012	
11.00	0.012	0.093
11.25	0.027	
11.5	0.016	0.13
11.75	0.041	
12.00	0.065	0.215
12.25	0.12	
12.5	0.093	0.215
12.75	0.081	
13.00	0.13	0.23
13.25	0.085	
13.5	0.14	0.26
13.75	0.13	
14.00	0.14	0.23
14.25	0.13	
14.5	0.15	0.25
14.75	0.12	
15.00	0.15	0.285
15.25	0.15	
15.5	0.12	0.27
15.75	0.12	
16.00	0.16	0.27

Run No.: 30R1

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:2

Catalyst No.: MSU KN C5(1st-reg.) Base: Nalco C
Metal, wt%: MoO_3=12.08(.1374) CoO = 0.00
NiO = 0.00 WO_3 = 0.00
( ) metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 89.06

Time vs. Nitrogen-Sulfur Content

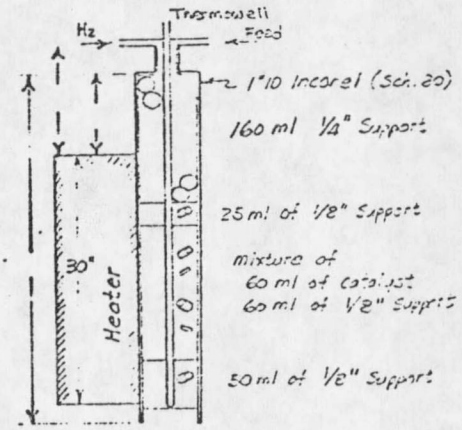
Min.	N	S	Min.	N	S
Feed	See the next page		135		
30			150		
45			165		
60			180		
75			195		
90			210		
105			225		
120			240		

ASTM Distillation
Volume of Charge, ml:

Vol.	IBP:382 F; 383 F
5	386 385
10	388 387
15	389 388
20	390 390
25	392 397
27	411
29	403
40	
45	
50	

Charge 35 cc 40 cc
1st 2hr last 2hr
composite composite

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_3;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 30R1

Hour	N	S	Hour	N	S
Feed	0.17	0.38	11.25	0.17	
0.5	0.00		11.5	0.16	0.32
0.75	0.00	0.18	11.75	0.18	
1.00	0.008		12.00	0.14	0.295
1.25	0.008		12.25	0.14	
1.5	0.024	0.23	12.5	0.16	0.305
1.75	0.016		12.75	0.14	
2.00	0.012	0.28	13.00	0.16	0.295
2.25	0.019		13.25	0.18	
2.5	0.02	0.33	13.5		0.335
2.75	0.02		13.75	0.17	
3.00	0.023	0.375	14.00	0.14	0.26
3.25	0.035		14.25	0.11	
3.5	0.038	0.24			
3.75	0.035				
4.00	0.038	0.205			
4.25	0.07				
4.5	0.043	0.20			
4.75	0.043				
5.0	0.043	0.205			
5.25	0.061				
5.5	0.047	0.225			
5.75	0.044				
6.00	0.043	0.24			
6.25	0.081				
6.5	0.13	0.305			
6.75	0.10				
7.00	0.10	0.285			
7.25	0.20				
7.5	0.14				
7.75	0.11				
8.00	0.09				
8.25	0.14				
8.5	0.16				
8.75	0.16				
9.00	0.16				
9.25	0.13				
9.5	0.16				
9.75	0.15				
10.00	0.13				
10.25	0.12				
10.5	0.21	0.32			
10.75	0.13				
11.00	0.15	0.335			

Run No.: 30R2

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C5 Base: Nalco C
Metal, wt%: MoO_3=12.08(.1374) CoO = 0.00
NiO = 0.00 WO_3 = 0.00
( ) metal oxide in gm per gm base

Run Condition
Temp., C : 425°
Press., psig: 1000
LHSV : 1.0
H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 88.44

Time vs. Nitrogen-Sulfur Content

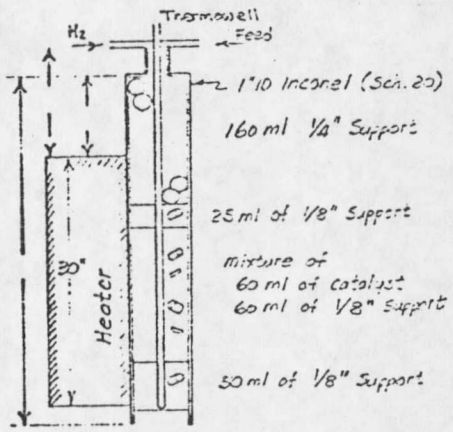
Min.	N	S	Min.	N	S
Feed	See the next page		135		
30			150		
45			165		
60			180		
75			195		
90			210		
105			225		
120			240		

ASTM Distillation

Volume of Charge, ml:

Vol.	IBP: 382 F; 381 F	
5	384	384
10	386	385
15	388	387
20	390	390
25	394	398
26.5		413
29.5	411	
40		
45		
50		
Charge	35 cc lst composite	40 cc last 2hr composite

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 31

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C11 Base: Nalco C
Metal, wt%: MoO_3= 4.26(.0491) CoO = 7.76(.0895)
NiO = 1.22(.0141) WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed:10,000 scf/bbl

Yield of Oil, Vol.%: 57.9

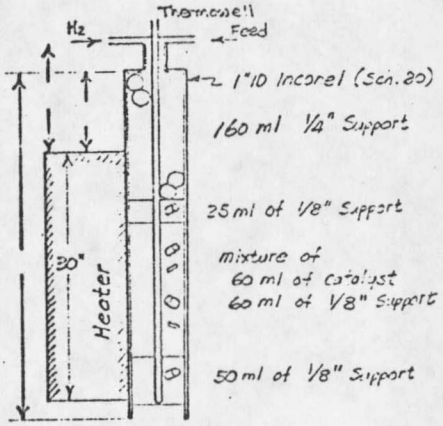
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.22	0.405	135		
30	0.01		150		
45	0.06		165		
60	0.03		180		
75	0.05		195		
90	0.01	0.965	210		
105	0.02		225		
120	0.01	0.495	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (369 °C)
5	380
10	381
15	384
20	386
25	388
30	391
32	392
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 32

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio=1:2

Catalyst No.: MSU KN C12 Base: Nalco C
Metal, wt%: MoO_3= 3.78(.0491) CoO = 6.90(.0895)
NiO = 1.08(.0141) WO_3 =11.16(.145)
( )metal oxide in gm per gm base

Run Condition
Temp., C : 430°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 69.3

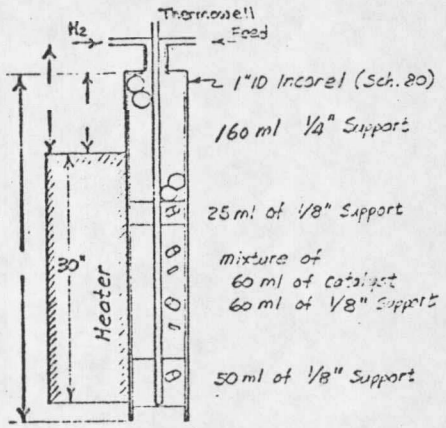
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.19	0.39	135		
30	0.01		150		
45	0.02		165		
60	0.01		180		
75	0.02		195		
90	0.01	0.295	210		
105	0.01		225		
120	0.01	0.29	240		

ASTM Distillation
Volume of Charge, ml:35.0

Vol.	IBP (309 °C)
5	370
10	376
15	378
20	382
25	385
30	394
32	395
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 33

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C13 Base: Nalco C
Metal, wt%: MoO_3=17.77(.2162) CoO = 0.
NiO = 0. WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 52.5

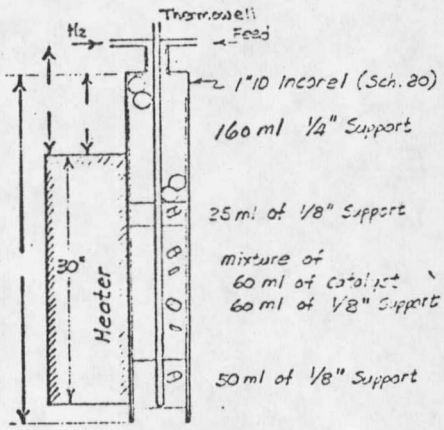
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.19	0.39	135		
30	0.01		150		
45	0.01		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.135	210		
105	0.01		225		
120	0.01		240		

ASTM Distillation
Volume of Charge, ml: 30.0

Vol.	IBP (371 °C)
5	380
10	383
15	385
20	388
25	394
27	397
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 34

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C14 Base: Nalco C
Metal, wt%: MoO_3=17.66(.2162) CoO = 0.
NiO = 0.64(.0078) WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 56.3

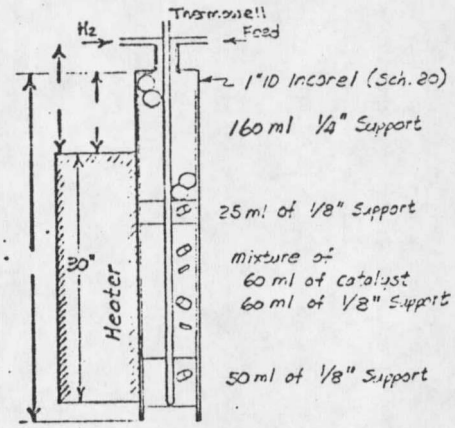
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.15	0.375	135		
30	0.01		150		
45	0.07		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.605	210		
105	0.02		225		
120	0.01	0.18	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (363 °C)
5	371
10	377
15	379
20	382
25	385
30	392
32	393
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 35

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C15 Base: Nalco C
Metal, wt%: MoO_3=16.88(.2162)CoO = 4.42(.0565)
NiO = 0.61(.0078)WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 60.0

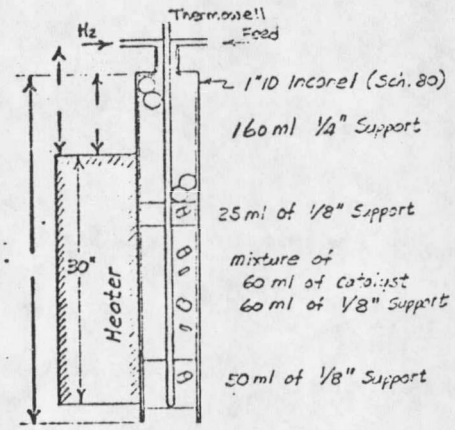
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.14	0.395	135		
30	0.07		150		
45	0.01		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.345	210		
105	0.01		225		
120	0.01	0.155	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (364 °C)
5	377
10	380
15	383
20	385
25	389
30	398
32	399
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 36

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C16 Base: Nalco C
Metal, wt%: MoO_3 = 15.30(.2162) CoO = 4.00(.0565)
NiO = 0.55(.0078) WO_3 = 9.39(.1328)
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 57.9

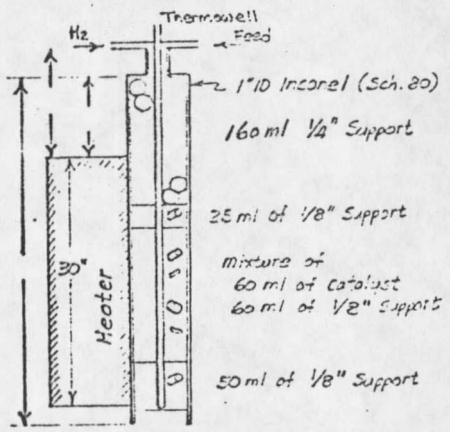
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.14	0.395	135		
30	0.01		150		
45	0.01		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.105	210		
105	0.02		225		
120	0.02	0.085	240		

ASTM Distillation
Volume of Charge, ml:35.0

Vol.	IBP (351 °C)
5	368
10	372
15	375
20	379
25	384
30	394
31	395
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 37

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C17 Base: Nalco C
Metal, wt%: MoO_3=15.36(.1815) CoO = 0.
NiO = 0. WO_3 = 0.
( ) metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 63.8

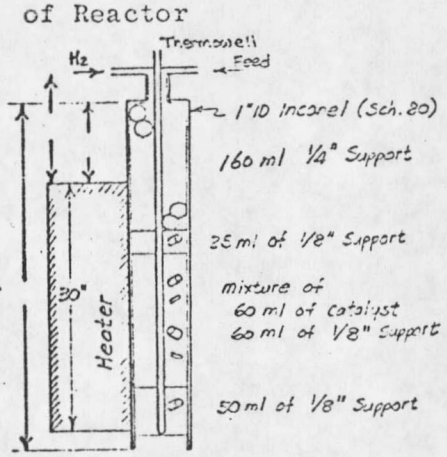
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.18	0.425	135		
30	0.03		150		
45	0.02		165		
60	0.01		180		
75	0.01		195		
90	0.01	1.68(?)	210		
105	0.01		225		
120	0.01	0.555	240		

ASTM Distillation
Volume of Charge, ml:35.0

Vol.	IBP (359 °C)
5	377
10	381
15	384
20	386
25	388
30	389
31	391
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 38

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C18 Base: Nalco C
Metal, wt%: MoO_3=15.11(.1815) CoO = 0.
NiO = 1.66(.0199) WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 69.6

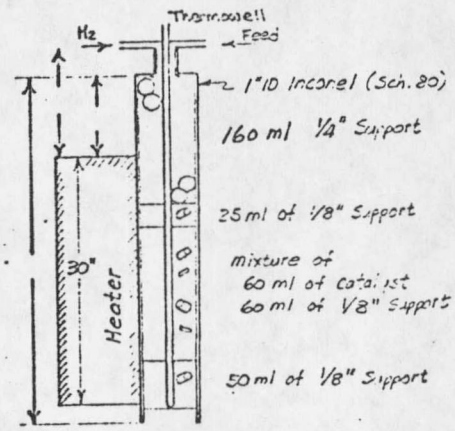
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.18	0.425	135		
30	0.01		150		
45	0.01		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.058	210		
105	0.01		225		
120	0.01	0.055	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (148 °C)
5	368
10	373
15	376
20	380
25	385
30	394
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m²/gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 39

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C19 Base: Nalco C
Metal, wt%: MoO_3=14.16(.1815) CoO = 6.23(.08)
NiO = 1.56(.0199) WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 57.0

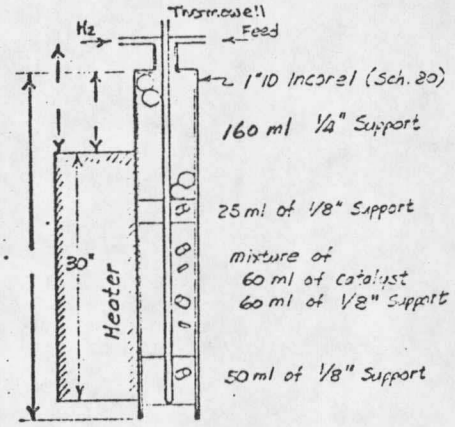
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.18	0.43	135		
30	0.01		150		
45	0.01		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.115	210		
105	0.01		225		
120	0.01	0.10	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (358 °C)
5	370
10	374
15	379
20	382
25	387
29.5	392
35	
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 40

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: MSU KN C20 Base: Nalco C
Metal, wt%: MoO_3=12.72(.1815) CoO = 5.60(.08)
NiO = 1.40(.0199) WO_3 =10.20(.1456)
( )metal oxide in gm per gm base

Run Condition
Temp., C : 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 60.5

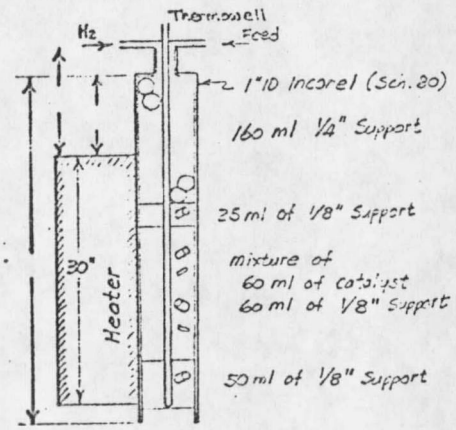
Time vs. Nitrogen-Sulfur Content

Min.	N	S	Min.	N	S
Feed	0.18	0.43	135		
30	0.03		150		
45	0.01		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.051	210		
105	0.01		225		
120	0.01	0.044	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (351 °C)
5	368
10	372
15	377
20	380
25	386
30	394
31	396
40	
45	
50	

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 41

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: Cyanamid HDS-20A Base: *
Metal, wt%: MoO_3 = 16.2 CoO = 5.0
NiO = 0. WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 440 - 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 58.2

Time vs. Nitrogen-Sulfur Content

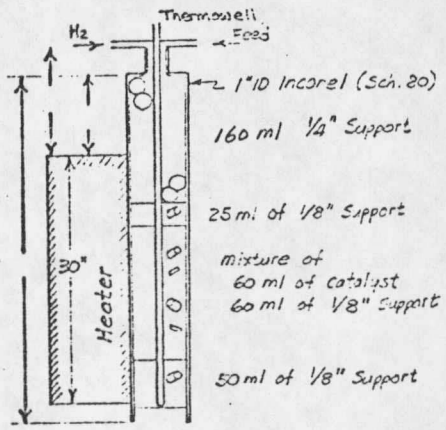
Min.	N	S	Min.	N	S
Feed	0.245	0.455	135		
30	0.014		150		
45	0.01		165		
60	0.02		180		
75	0.032		195		
90	0.01	0.081	210		
105	0.01		225		
120	0.01	0.044	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (347 °C)
5	367
10	373
15	379
20	384
25	389
30	395
35	
40	
45	
50	

* SA: 230 m^2/gm
PV: .52 cc/gm

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2 ; 96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2 ; 98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5 ; 91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5; 83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

Run No.: 42

Feed Material: Solvent Refined Lignite (SRL)
Solvent : Tetralin SRL to Tetralin ratio= 1:2

Catalyst No.: SHELL 324 Base: *
Metal, wt%: MoO_3= 13.0 CoO = 0.
NiO = 3.0 WO_3 = 0.
( )metal oxide in gm per gm base

Run Condition
Temp., C : 440 - 425°C LHSV : 1.0
Press., psig: 1000 H_2:Feed: 10,000 scf/bbl

Yield of Oil, Vol.%: 61.8

Time vs. Nitrogen-Sulfur Content

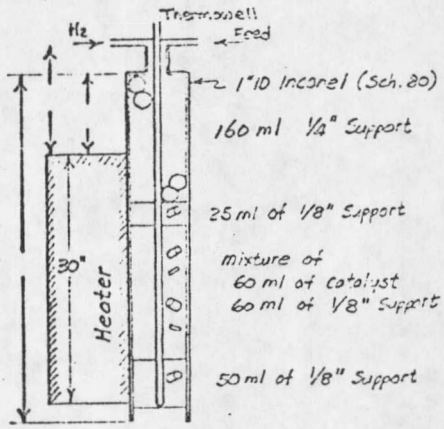
Min.	N	S	Min.	N	S
Feed	0.245	0.455	135		
30	0.01		150		
45	0.01		165		
60	0.01		180		
75	0.01		195		
90	0.01	0.053	210		
105	0.01		225		
120	0.01	0.045	240		

ASTM Distillation
Volume of Charge, ml: 35.0

Vol.	IBP (345 °C)
5	365
10	371
15	376
20	382
25	389
30	395
31	403
40	
45	
50	

* SA: m^2/gm
PV: cc/gm

Cross-sectional View of Reactor



Characteristics of Nalco Catalyst Bases

Nalco Base No.	Composition	Surface Area m /gm	Pore Vol. ml/gm	Median Pore D. A°	Average Pore D. A° *
A (1/32") 78-6008	4%SiO_2;96%Al_2O_3	323.2	0.7183	90.2	88.9
B (1/16") 78-6008	100%Al_2O_3	232.4	0.7215	137.3	124.2
C (1/32") 78-6008	2%SiO_2;98%Al_2O_3	214.6	0.8397	161.0	156.5
D (1/16") 78-6008	9%P_2O_5;91%Al_2O_3	211.4	0.7943	190.0	150.3
E (1/16") 78-6008	17%P_2O_5;83%Al_2O_3	147.0	0.6841	420.2	186.2

*Average Pore Diameter (A) = 40,000 (pore volume/surface area)

A Computer Program to Predict the %DN
As a Function of Time on Stream:Run 30R2

```
1.000 C     THIS IS TO FIND TWO CONSTANTS FOR A MODEL
2.000 C     MADE TO PREDICT THE DENITROGENATION AS A
3.000 C     FUNCTION OF TIME

4.000       DIMENSION X(5,20),XL(5),XH(5),XC(5),XX(5),FUNC(20),
5.000      &XP(5,20),XXP(5)
6.000       DIMENSION EXDN(20),CALDN(20)
7.000       COMMON CALDN
7.500       DATA EXDN/92.5,96.3,94.4,89.4,78.6,74.7,68.2,51.3,
7.500*     &34.4,37.2,31.9,18.8,12.6,18.8,9.4/
8.000 1     INPUT M,N,KK,KPRINT,EPSI
9.000       INPUT X(I,1),I=1,N
10.000      INPUT XL(I),I=1,N
11.000      INPUT XH(I),I=1,N
12.000      CALL CMBOX(M,N,KK,KPRINT,X,XL,XH,XC,XX,XP,XXP,
13.000     &FUNC,EPSI)
14.000      DO 50 J=1,15
15.000      WRITE (108,200)J,EXDN(J),CALDN(J)
16.000 200  FORMAT(3G12.5)
17.000 50   CONTINUE
18.000      GO TO 1
19.000      END
```

```
20.000          SUBROUTINE OBJ(X,N,XP,OB,KOB)
21.000          DIMENSION X(N),XP(N)
22.000          DIMENSION EXDN(20),CALDN(20)
24.000          COMMON CALDN
25.000          DATA EXDN/92.5,96.3,94.4,89.4,78.8,74.7,68.2,51.3,
26.000         &34.4,37.2,31.9,18.8,12.6,18.8,9.4/
27.000          DEL=0.
28.000          KOB=KOB+1.
29.000          DO 100 J=1,15
30.000          CALDN(J)=100.-100./(1.+X(1)*X(2)**J)
31.000          DEL=(EXDN(J)-CALDN(J))**2+DEL
32.000 100      CONTINUE
33.000          OB=-DEL
34.000          DO 2 I=1,N
35.000 2        XP(I)=X(I)
36.000          RETURN
37.000          END
38.000          SUBROUTINE CONSTR(X,N,IVI)
39.000          DIMENSION X(N)
40.000          IVI=0
41.000          G1=X(1)
42.000          IF(G1.LT.0.) IVI=1;RETURN
43.000          G2=X(2)
44.000          IF(G2.LT.0.) IVI=1;RETURN
45.000          G3=10000.-X(1)
46.000          IF(G3.LT.0.) IVI=1;RETURN
47.000          G4=1.-X(2)
48.000          IF(G4.LT.0.) IVI=1
49.000          RETURN
50.000          END
```

```
?1,2,4,0,.0000001
?1000.,.5
?0.,0.
?10000.,1.
  -491.18      -457.55      -497.57      -513.79
   57.951       48.920       59.139       68.836
   .61502       .62508       .63084       .61265

 FUNCTIONAL VALUES WITHIN FRACTIONAL DIFFERENCE =   .78907E-06
  177 CYCLES WITH   4 VERTICES HAVE BEEN COMPUTED
 OBJECTIVE HAS BEEN EVALUATED  304 TIMES
  -309.40      -309.40      -309.40      -309.40   Σ_i (EXP_i - CALC_i)^2
   29.774       29.772       29.765       29.770   A
   .67140       .67141       .67142       .67141   B

                EXP %DN      CALC %DN
     1          92.500       95.295
     2          86.300       93.065
     3          84.400       90.010
     4          89.400       85.915
     5          78.800       80.244
     6          74.700       73.170
     7          68.200       64.677
     8          51.300       55.144
     9          34.400       45.217
    10          37.200       35.857
    11          31.900       27.118
    12          18.800       19.889
    13          12.800       14.384
    14          18.800       10.122
    15          9.4000       7.0296
```

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Kim, Nam K
Catalysts for hydro-
treating solvent refined
lignite

DATE	ISSUED TO
4 MAY 87	Gary R Peterson ChE Dept 222 (G) MDS

[redacted]

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