

CATALYTIC HYDROGENATION OF COAL-DERIVED LIQUIDS

Interim Report for the Period

June 1980 - August 1980

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PROJECT TITLE: CATALYTIC HYDROGENATION OF COAL-DERIVED LIQUIDS
Contract No. EX-76-C-01-2034

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SUMMARY

It is the objective of this research to convert coal to clean distillate fuels. This program will be limited to research on the product of PAMCO's solvent refined coal (SRC-II). The SRC will be heated and pumped, with and without solvent, into a catalytic reactor in the presence of hydrogen and other reducing gases. Variables to be investigated will include temperature, pressure, space velocity, hydrogen-to-oil ratio and chemical nature of the solvent. The catalysts to be studied will include nickel molybdate and sulfide, nickel tungstate and other combinations on carriers such as mordanites and other molecular sieve types.

During this quarter sixteen catalysts comprising each possible four metal combination of 2 and 4% CoO, 2 and 8% MoO₃, 1 and 4% NiO and 2 and 8% WO₃ were fabricated on the same base material, tested with SRC-II and the results analysed statistically. Only a change in the concentration of CoO had a significant effect and this was small.

Analyses of products were completed on the evaluation of the non-promoted base material as well as the T.C.C. cracking catalyst. They confirmed the conclusion stated in our previous report that these catalysts are incapable of effecting sufficient denitrogenation to meet our goals.

All of these catalysts gave significant denitrogenation;

OBJECTIVE

It is the objective of this research to convert coal to clean distillate fuels. This program will be limited to research on the product of PAMCO's solvent refined coal (SRC-II). The SRC will be heated and pumped, with and without solvent, into a catalytic reactor in the presence of hydrogen and other reducing gases. Variables to be investigated will include temperature, pressure, space velocity, hydrogen-to-oil ratio and chemical nature of the solvent. The catalysts to be studied will include nickel molybdate and sulfide, nickel tungstate and other combinations on carriers such as mordanites and other molecular sieve types.

ABSTRACT

The work on the development of catalysts capable of converting solvent refined coal, SRC-II, into a feedstock suitable for a conventional petroleum refinery was continued. This requires a process which reduces the nitrogen content from the 1.17% in SRC-II to at least as low as 0.3%.

During this quarter sixteen catalysts were fabricated and evaluated in a statistical study. They comprised every combination of 2 and 4% CoO, 2 and 8% MoO₃, 1 and 4% NiO and 2 and 8% WO₃ on an alumina base possessing a surface area of 214 m²/gram and a pore diameter of 156Å. All of them gave specification denitrogenation and CoO was the only one of the four metal oxides that caused a statistical change in denitrogenation capability.

CONVERSION OF SOLVENT REFINED COAL, SRC-II, TO DISTILLATE FUELS,
By An-gong Yeh

The purpose of this research is to develop new catalysts capable of converting PAMCO's Solvent Refined Coal, SRC-II, into clean distillate fuels and/or a feedstock acceptable to a conventional petroleum refinery. To accomplish this, the nitrogen content of the SRC-II which is 1.17 wt.%, must be reduced as much as possible but at least to 0.3%. The sulfur content, 0.72 wt.%, should be reduced as much as possible also but denitrogenation is considered by us to be the major problem.

In this research SRC-II is catalytically hydrotreated in a trickle bed reactor. The reactor is made by a one-inch I.D. 40 inches long Schedule 80 Inconel pipe. The arrangement of equipment is shown in Figure 1. Hydrogen and SRC-II are fed at the top of reactor. Starting from the top, the reactor is loaded with 175 ml. of 1/4" Denstone inert support, 25 ml. of 1/8" Denstone inert support, followed by a mixture of 60 ml. of catalyst and 60 ml. of 1/8" Denstone inert support. The remaining space at the bottom of the reactor is filled with 1/8" Denstone inert support. Operating conditions usually are 425°C, 1,000 psig, liquid hourly velocity of 1.0, hydrogen feed rate of 10,000 scf/bbl of oil, and SRC-II feed temperature of 85°C.

Previously, the non-metal-loaded base material, Nalco-78-6008C-1/32", possessing a surface area of 214.6 m²/gram, a pore volume of 0.839 ml./gram, a pore diameter of 156.5 Å, and a median pore diameter of 161 Å was tested at different SRC-II feed temperatures ranging from 85°C to 200°C in Runs A-31 to A-34. Table I summarizes the results of blank runs for denitrogenation and desulfurization. The more detailed data is included in Appendix A. The percent of denitrogenation obtained from Runs A-31 and A-34 are regressed on the running time in order to compare with that obtained from runs using the metal loaded catalysts. The regression equation

$$\%DN = 70.5 e^{-0.01t} \quad (1)$$

with a R-square of 0.70 seems appropriate to fit the data, where the weight percent of denitrogenation %DN is calculated by $(1.17\% - \text{wt\%})/1.17\%$ and t represents the running time in minutes. The experimental data and the fitted regression line are plotted in Figure 2. The table of the analysis of variance (ANVOA) is shown in Appendix B.

The T.C.C. equilibrium cracking catalyst obtained from the Mobil Oil Corporation was tested in Runs A-35 and A-36. In Run A-35 the SRC-II was diluted with two parts of tetralin in order to make the pumping easier. Run A-36 was fed with

undiluted SRC-II at 100°C. The results for denitrogenation have been reported before, which is less than 50%. The results for desulfurization shown in Appendix A have reached 80%.

During the quarter, sixteen catalysts have been developed by impregnating the base material, Nalco-78-6008C-1/32", with CoO, MoO₃, NiO, and WO₃. A 2⁴ experimental design was set up by choosing two different levels for each metal oxide, 2% and 8% for CoO, 2% and 8% for MoO₃, 1% and 4% for NiO, 2% and 8% for WO₃. Runs A-41 to A-56 shown in Appendix A tested the catalysts with these combinations. Tables II and III list the results of denitrogenation and desulfurization, respectively. The data is analyzed by the forward stepwise regression technique to investigate the variance and the interactions between metal oxides.

The weight percent of denitrogenation in Table II is regressed on the running time and the compositions of catalysts. The results show CoO gives a positive effect on the denitrogenation in which the denitrogenation is expected to increase by 3.5% when the amount of CoO increases by 1 wt%. The main effects and the interactions of MoO₃, NiO, and WO₃ are insignificant at the level of significance of 0.05. The experimental data and fitted regression line are plotted in Figure 2. By comparing with the denitrogenation of non-metal-loaded base material, catalysts with metal oxides did give a higher activity on nitrogen removal. The R-square of the regression equation appears to 0.81 and ANOVA table is shown in Appendix C. The variance of average denitrogenation in 2.5 hours between two levels of CoO is plotted in Figure 3.

The average weight percent of desulfurization in 2.5 hours shown in Table II is regressed on the composition of catalyst. It is observed that the effects of CoO, MoO₃, and WO₃ on desulfurization are significant by using a confidence coefficient of 95 percent. The average desulfurization is expected to decrease by 8.5% when the amount of CoO increases by 1 wt%, to increase by 3.5% when the amount of MoO₃ increases by 1 wt%, and to increase by 1.6% when the amount of WO₃ increases by 1 wt%. Figures 4, 5, and 6 plot the variances between two levels for CoO, MoO₃, and WO₃. The effect of NiO and the interactions between metal oxides are found to be insignificant. The ANOVA table is shown in Appendix D and the R-square of the regression equation is 0.81.

In summary, a sixteen point statistical combination of concentrations each of CoO, MoO₃, NiO and WO₃ all on the same base material, showed rather little variability in denitrogenation and desulfurization ability. Only the change in the concentration of CoO seemed to be significant and this was small.

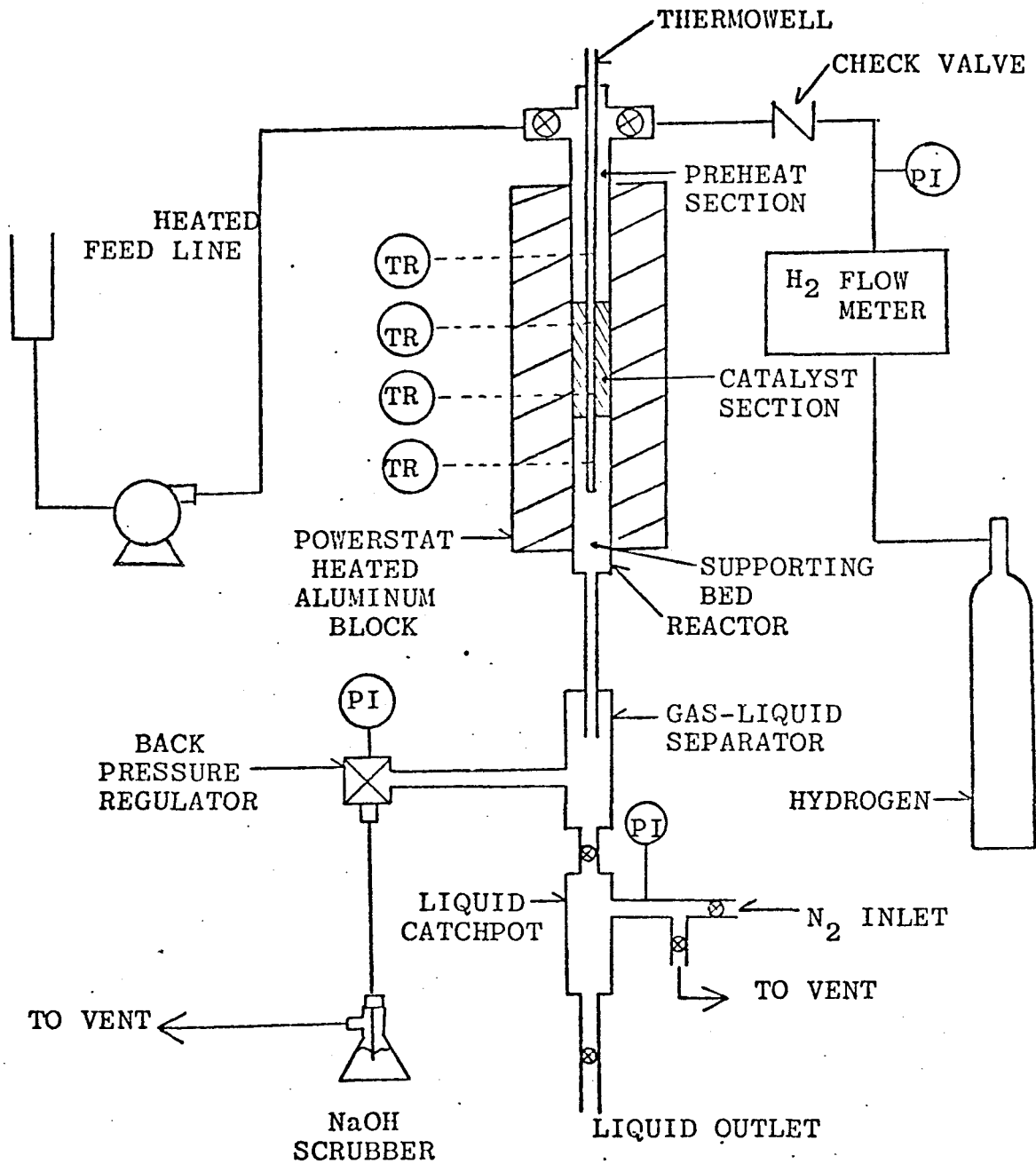


FIGURE 1. TRICKLE BED REACTOR

TABLE I BLANK RUNS* DATA SUMMARY

Run	%DN						%DS		SRC-II Feed Temperature °C
	Time, min.						Time, min.		
	45	60	75	90	105	120	90	120	
A-31	65.4	39.7	35.9	27.4	26.6	18.8	11.8	32.6	85
A-32	73.5	39.3	28.2	23.1	39.3	37.6	26.4	20.1	105
A-33	77.8	-----coke up-----					55.6**	-	200
A-34	40.2	27.8	25.6	27.4	23.5	23.5	24.3	31.1	85

* Non-metal-loaded base material is Nalco-78-6008C-1/32".

** Sample collected up to 45 minutes.

TABLE II
DENITROGENATION FOR 2⁴ EXPERIMENTAL DESIGN

Run	%DN								Metal Oxides Loaded on the Base Material*, wt%			
	Time, min.								CoO	MoO ₃	NiO	WO ₃
	45	60	75	90	105	120	150	average				
A-41	92.0	89.3	85.5	77.8	72.6	61.5	50.0	75.5	2	8	1	8
A-42	85.5	92.8	69.2	61.5	48.7	44.0	35.9	62.5	2	8	1	2
A-43	97.0	96.2	91.0	80.3	65.4	56.4	51.7	76.8	2	8	4	8
A-44	98.6	92.2	80.8	68.8	51.3	42.7	43.6	68.3	2	8	4	2
A-45	98.3	87.2	79.5	62.8	56.4	45.3	30.3	65.7	2	2	1	8
A-46	93.8	79.5	65.8	57.7	49.1	44.9	42.3	61.9	2	2	1	2
A-47	95.5	89.7	81.6	66.7	58.5	52.1	35.9	68.6	2	2	4	8
A-48	93.2	80.8	70.5	64.1	57.7	54.3	48.7	67.0	2	2	4	2
A-49	98.6	93.8	88.9	80.3	66.7	60.7	56.8	77.9	4	8	1	8
A-50	97.3	91.7	84.6	78.6	69.7	60.3	57.3	77.1	4	8	1	2
A-51	97.9	92.1	81.2	70.9	62.0	55.6	57.3	73.8	4	8	4	8
A-52	89.7	87.2	82.0	72.2	68.4	66.7	50.8	73.8	4	8	4	2
A-53	95.1	88.9	79.1	70.1	57.7	52.6	57.3	71.5	4	2	1	8
A-54	92.3	99.1	97.4	73.5	71.8	70.1	63.2	81.1	4	2	1	2
A-55	97.4	88.6	70.1	75.2	72.6	68.4	57.3	75.6	4	2	4	8
A-56	94.9	91.4	79.5	58.1	91.4	47.0	28.2	70.1	4	2	4	2

* Base material is Nalco-78-6008C-1/32".

1
8
1

TABLE III
DESULFURIZATION FOR 2⁴ EXPERIMENTAL DESIGN

Run	Average %DS	Metal Oxides Loaded on the Base Material*, wt%			
		CoO	MoO ₃	NiO	WO ₃
A-41	77.4	2	8	1	8
A-42	63.9	2	8	1	2
A-43	67.4	2	8	4	8
A-44	70.8	2	8	4	2
A-45	64.9	2	2	1	8
A-46	34.4	2	2	1	2
A-47	46.5	2	2	4	8
A-48	41.8	2	2	4	2
A-49	47.6	4	8	1	8
A-50	43.1	4	8	1	2
A-51	56.2	4	8	4	8
A-52	55.9	4	8	4	2
A-53	41.0	4	2	1	8
A-54	34.7	4	2	1	2
A-55	36.8	4	2	4	8
A-56	14.9	4	2	4	2

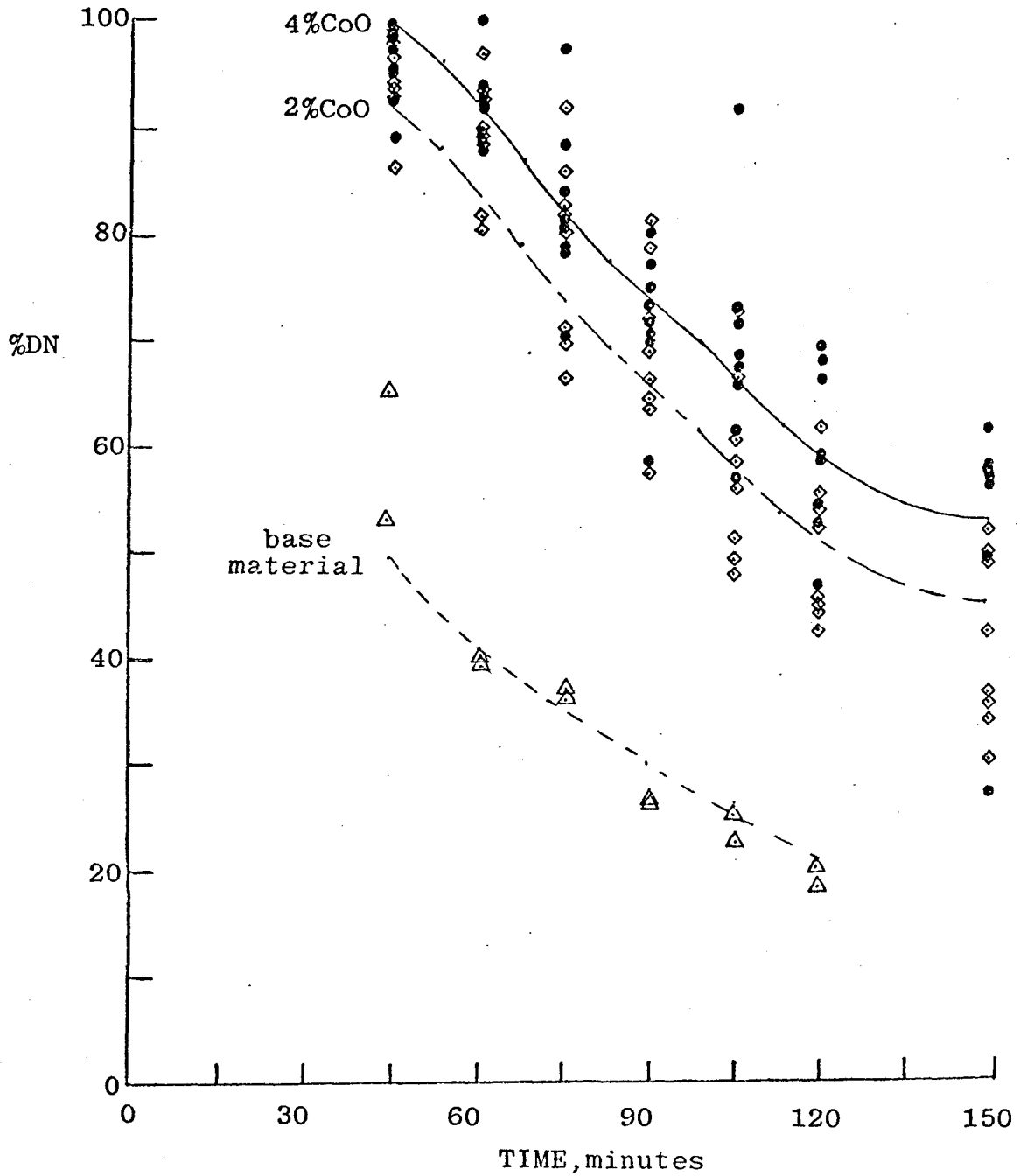


Figure 2. Nitrogen removal as a function of running time and CoO concentration.

● 4%CoO

◇ 2%CoO

△ base material Nalco-78-6008C-1/32"

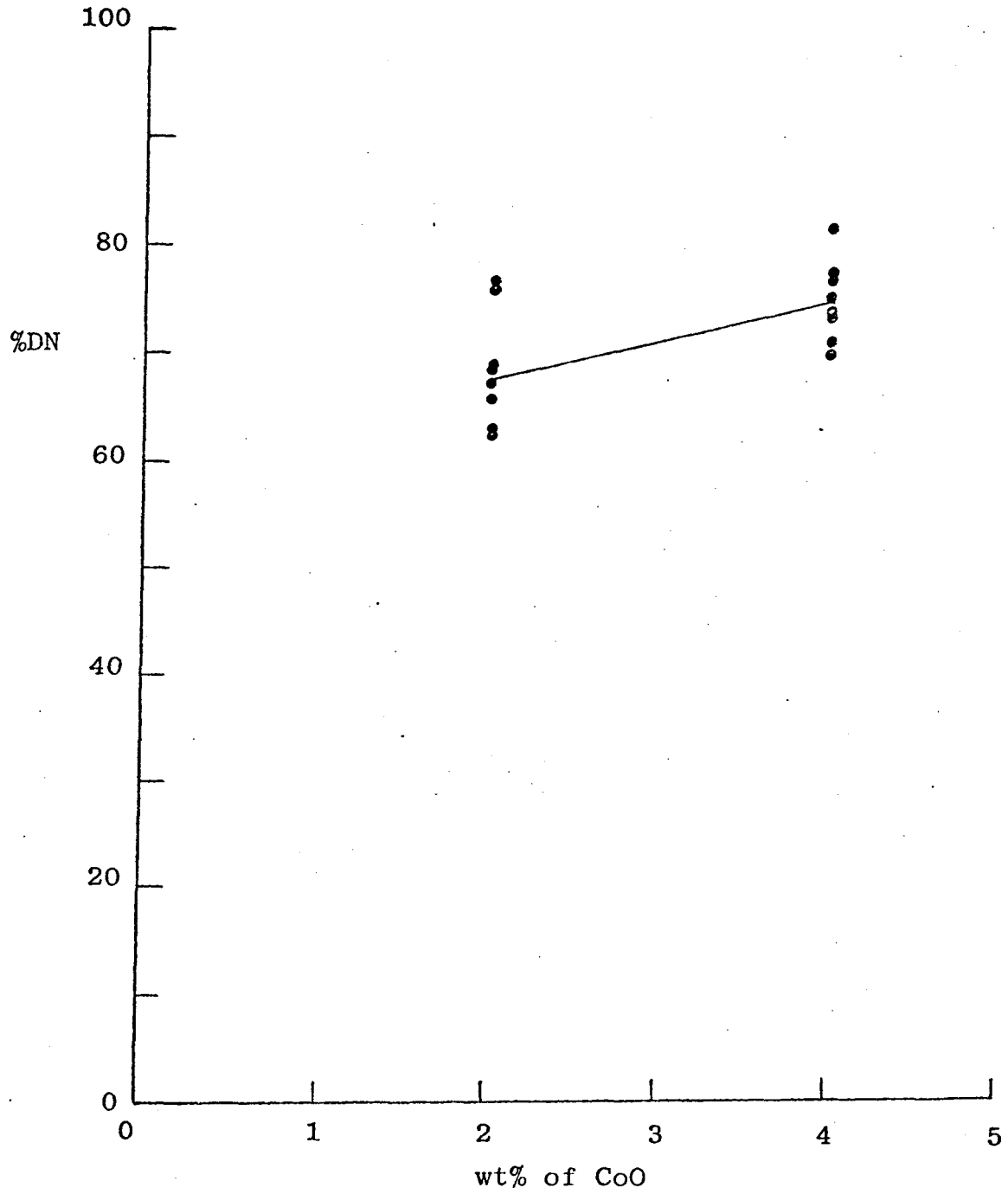


Figure 3. The effect of CoO concentration on denitrogenation.

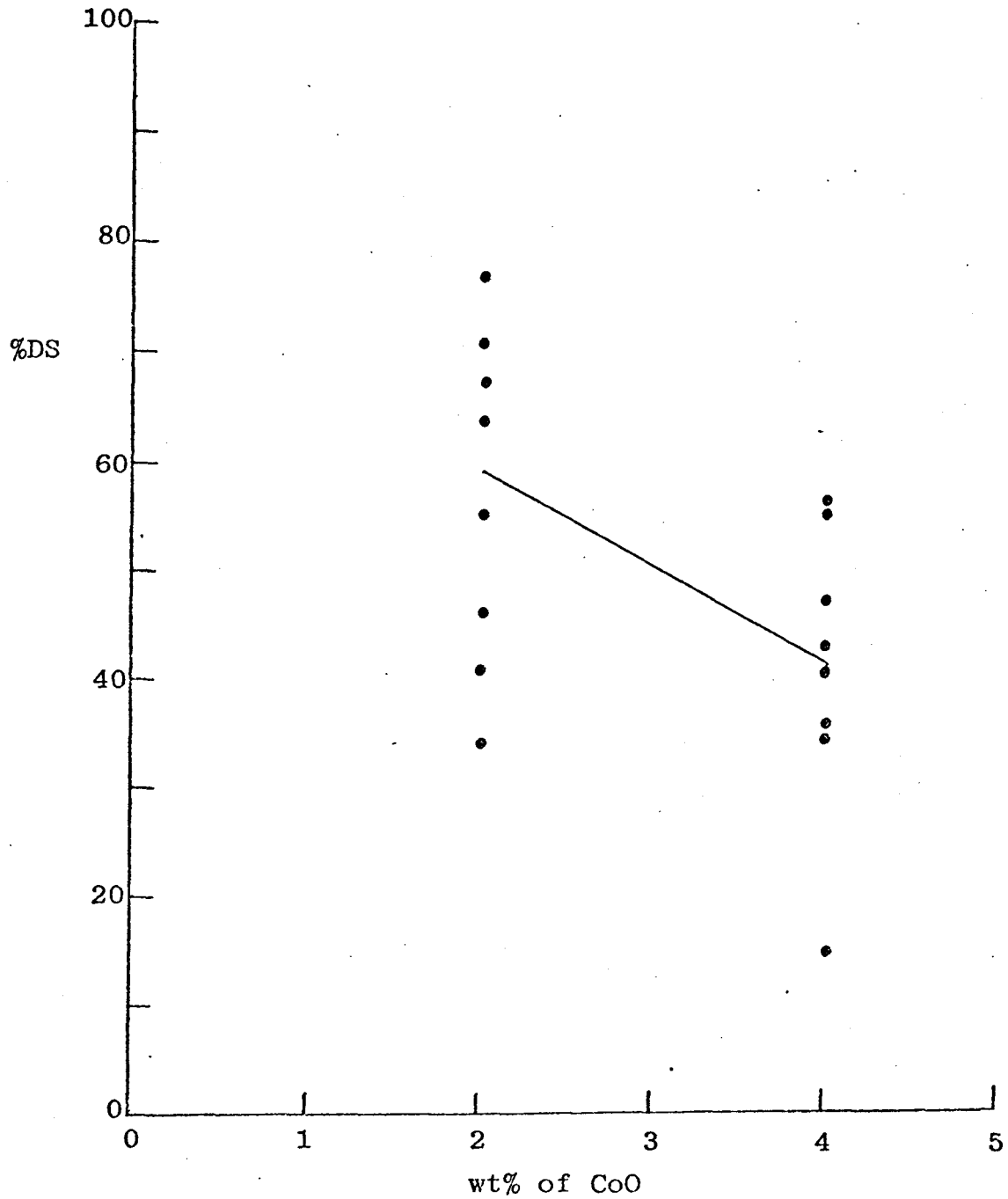


Figure 4. The effect of CoO concentration on desulfurization.

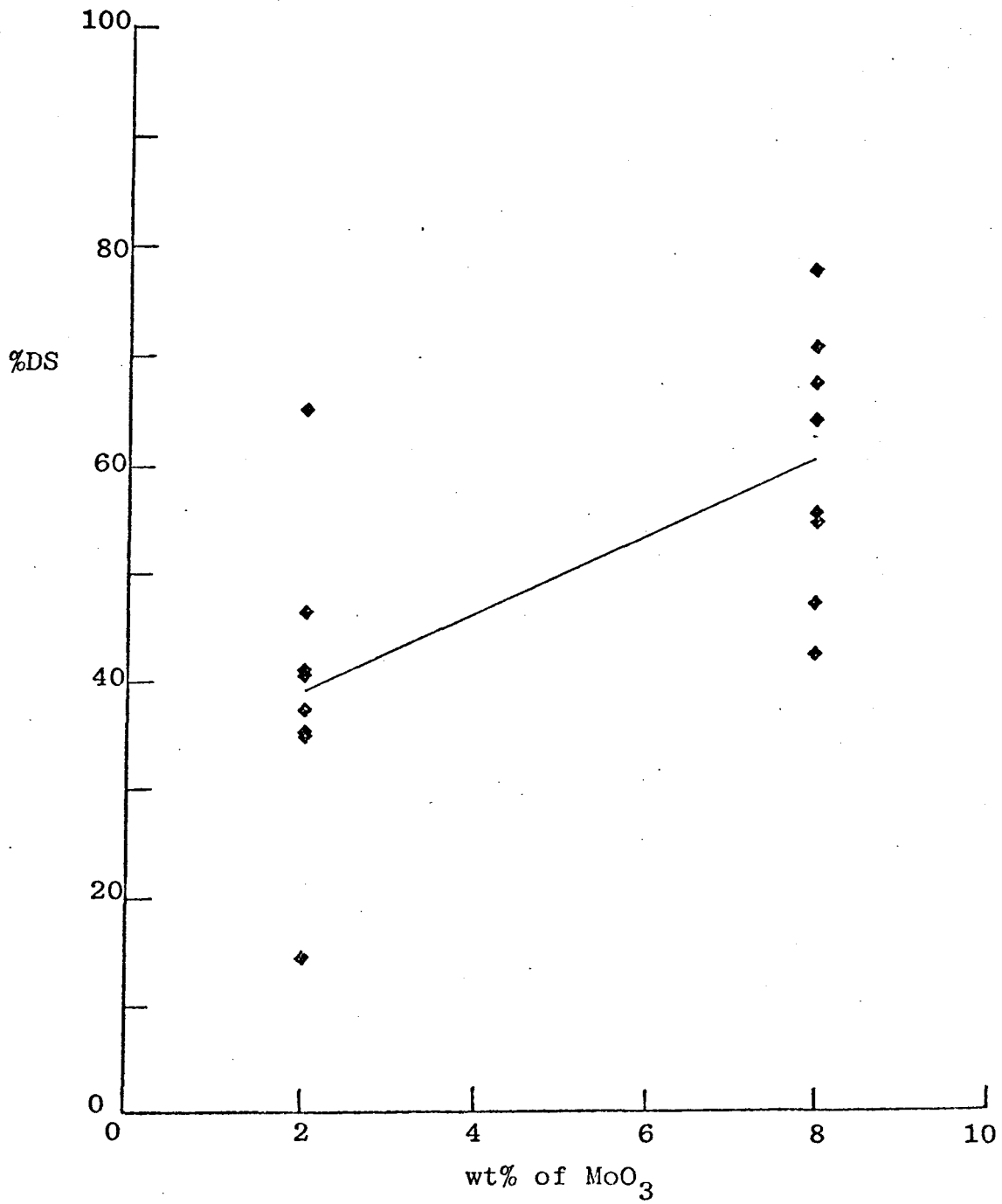


Figure 5. The effect of MoO₃ concentration on desulfurization.

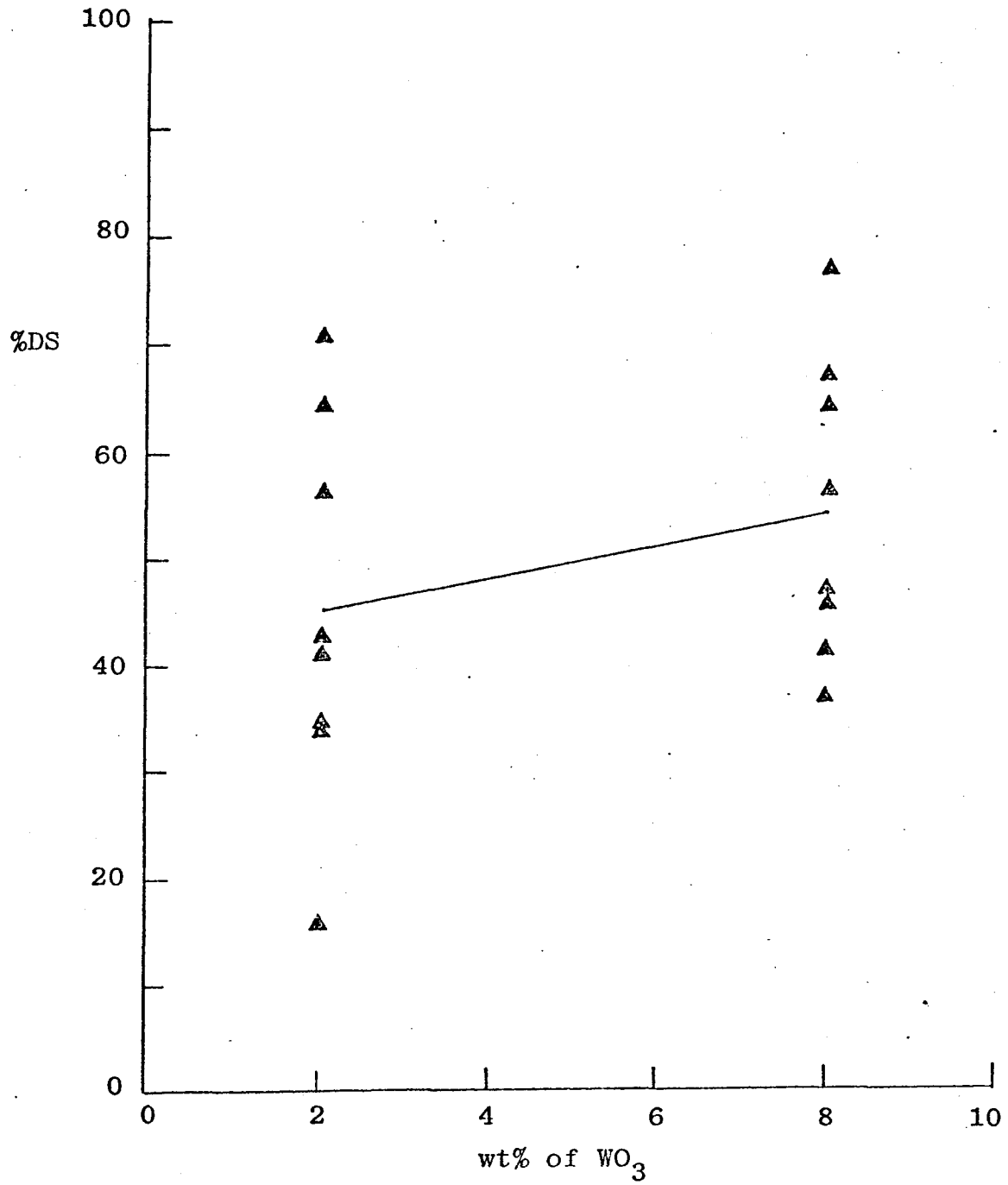


Figure 6. The effect of WO₃ concentration on desulfurization.

APPENDIX A

Run No. A-31

Catalyst No. NALCO-78-6008C-1/32"

Catalyst Composition

Metals : None

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

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

Run Temperature, °C : 425±5 SRC-II Feed Temperature, °C : 85

Run Pressure, psig : 1,025

Liquid Hourly Space Velocity : 1.1

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 5%

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

wt% N₂ : .46 .405 .705 .75 .85 .86 .95

wt% S : - - - - .635 - .485

ASTM Distillation

Volume of Charge: Final Volume:

Volume % : IBP 10 20 30 40 50
°F :
Volume % : 60 70 80 90
°F :

Run No. A-32

Catalyst No. NALCO-78-6008C-1/32"

Catalyst Composition

Metals : None

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

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

Run Temperature, °C : 425[±]5 SRC-II Feed Temperature, °C : 105

Run Pressure, psig : 1.025

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 73%

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

wt% N₂ : .25 .31 .71 .84 .90 .71 .73

wt% S : - - - - .53 - .575

ASTM Distillation

Volume of Charge: Final Volume:

Volume % : IBP 10 20 30 40 50
°F :
Volume % : 60 70 80 90
°F :

Run No. A-33

Catalyst No. NALCO-78-6008C-1/32"

Catalyst Composition:

Metals : None

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

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

Run Temperature, °C : 425[±]5 SRC-II Feed Temperature, °C : 200

Run Pressure, psig : 1,050

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

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

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

wt% N₂ : .12 .26

wt% S : - .11

ASTM Distillation

Volume of Charge: Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-34

Catalyst No. NALCO-78-6008C-1/32"

Catalyst Composition

Metals : None

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

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

Run Temperature, °C : 425[±]5 SRC-II Feed Temperature, °C : 85

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 44%

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

wt% N₂ : .355 .70 .845 .87 .85 .895 .895

wt% S : - - - - .545 - .496

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-35

Catalyst No. T.C.C. equilibrium catalyst

Catalyst Composition

Metals :

Base :

Base Surface Area, m²/g :

Base Pore Volume, ml/g :

Base Pore Diameter (4V/A), Å :

Base Median Pore Diameter, Å :

Run Temperature, °C : 425±5 SRC-II Feed Temperature, °C : 100

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 81%

Time, min:	30	45	60	75	90	105	120	150
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wt% N ₂ :	.07	.12	.09	.125	.165	.08	.19
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wt% S :	-	-	-	-	.05	-	.10
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ASTM Distillation

Volume of Charge:

Final Volume:

Volume % :	IBP	10	20	30	40	50
°F :						

Volume % :	60	70	80	90
°F :				

Run No. A-36

Catalyst No. T.C.C. equilibrium catalyst

Catalyst Composition

Metals :

Base :

Base Surface Area, m²/g :
Base Pore Volume, ml/g :
Base Pore Diameter (4V/A), Å :
Base Median Pore Diameter, Å :

Run Temperature, °C : 425±5 SRC-II Feed Temperature, °C : 100

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

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

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

wt% N₂ : .41 .50 .66 .56 .75 .64 .695

wt% S : - - - - .115 - .23

ASTM Distillation

Volume of Charge: Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-41

Catalyst No. C-41

Catalyst Composition

Metals : CoO 2% MoO₃ 8% NiO 1% WO₃ 8%

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 428 SRC-II Feed Temperature, °C : 90

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .92

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 72

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

wt% N₂ : - .093 .125 .170 .260 .320 .450 .585

wt% S : - - - - .110 - - .215

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-42

Catalyst No. C-42

Catalyst Composition

Metals : 2%CoO 8%MoO₃ 1%NiO 2%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 428 SRC-II Feed Temperature, °C : 92

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .99

H₂: Oil Ratio, scf/bbl : 10,000

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

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

wt% N₂ : - .170 .084 .360 .450 .600 .655 .750

wt% S : - - - - .290 - - .230

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-43

Catalyst No. C-43

Catalyst Composition

Metals : 2%CoO 8%MoO₃ 4%NiO 8%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 427 SRC-II Feed Temperature, °C : 92

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 62

Time, min:	30	45	60	75	90	105	120	150
wt% N ₂	-	.035	.044	.105	.230	.405	.510	.565
wt% S	-	-	-	-	.255	-	-	.215

ASTM Distillation

Volume of Charge:		Final Volume:				
Volume %	: IBP	10	20	30	40	50
°F	:					
Volume %	: 60	70	80	90		
°F	:					

Run No. A-44

Catalyst No. C-44

Catalyst Composition

Metals : 2%CoO 8%MoO₃ 4%NiO 2%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 434

SRC-II Feed Temperature, °C : 88

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.1

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 70

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

wt% N₂ : - .016 .091 .225 .365 .570 .670 .660

wt% S : - - - - .190 - - .230

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-45

Catalyst No. C-45

Catalyst Composition

Metals : 2%CoO 2%MoO₃ 1%NiO 8%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 422 SRC-II Feed Temperature, °C : 80

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 64

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

wt% N₂ : - .020 .150 .240 .435 .510 .640 .815

wt% S : - - - - .275 - - .230

ASTM Distillation

Volume of Charge: Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-46

Catalyst No. C-46

Catalyst Composition

Metals : 2% CoO 2%MoO₃ 1%NiO 2%WO₃

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

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

Run Temperature, °C : 425 SRC-II Feed Temperature, °C : 88

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .83

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 76

Time, min:	30	45	60	75	90	105	120	150	
wt% N ₂	:	-	.072	.240	.400	.495	.595	.645	.675
wt% S	:	-	-	-	-	.720	-	-	.225

ASTM Distillation

Volume of Charge:		Final Volume:				
Volume %	: IBP	10	20	30	40	50
°F	:					
Volume %	: 60	70	80	90		
°F	:					

Run No. A-47

Catalyst No. C-47

Catalyst Composition

Metals : 2%CoO 2%MoO₃ 4%NiO 8%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 429 SRC-II Feed Temperature, °C : 88

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 76

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

wt% N₂ : - .053 .120 .215 .390 .485 .560 .750

wt% S : - - - - .470 - - .300

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-48

Catalyst No.C-48

Catalyst Composition

Metals : 2%CoO 2%MoO₃ 4%NiO 2%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 424 SRC-II Feed Temperature, °C : 80

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .93

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 75

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

wt% N₂ : - .080 .225 .345 .420 .495 .535 .600

wt% S : - - - - .593 - - .245

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-49

Catalyst No. C-49

Catalyst Composition

Metals : 4%CoO 8%MoO₃ 1%NiO 8%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 425 SRC-II Feed Temperature, °C : 90

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : 1.0

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 76

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

wt% N₂ : - .016 .073 .130 .230 .390 .460 .505

wt% S : - - - - .495 - - .260

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-50

Catalyst No. C-50

Catalyst Composition

Metals : 4%CoO 8%MoO₃ 1%NiO 2%WO₃

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

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

Run Temperature, °C : 422 SRC-II Feed Temperature, °C : 82

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .88

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 74

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

wt% N₂ : - .032 .097 .180 .250 .355 .465 .500

wt% S : - - - - .420 - - .400

ASTM Distillation

Volume of Charge: Final Volume:

Volume % : IBP 10 20 30 40 50
°F :
Volume % : 60 70 80 90
°F :

Run No. A-51

Catalyst No. C-51

Catalyst Composition

Metals : 4%CoO 8%MoO₃ 4%NiO 8%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 420 SRC-II Feed Temperature, °C : 85

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .97

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 79

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

wt% N₂ : - .024 .092 .220 .340 .445 .420 .500

wt% S : - - - - .385 - - .245

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-52

Catalyst No. C-52

Catalyst Composition

Metals : 4%CoO 8%MoO₃ 4%NiO 2%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 420 SRC-II Feed Temperature, °C : 85

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .90

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 84

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

wt% N₂ : - .120 .150 .210 .325 .370 .390 .575

wt% S : - - - - .390 - - .245

ASTM Distillation

Volume of Charge: Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-53

Catalyst No. C-53

Catalyst Composition

Metals : 4%CoO 2%MoO₃ 1%NiO 8%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 420 SRC-II Feed Temperature, °C : 85

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .91

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 75

Time, min:	30	45	60	75	90	105	120	150	
wt% N ₂	:	-	.057	.130	.245	.350	.495	.555	.500
wt% S	:	-	-	-	.600	-	-	.250	

ASTM Distillation

Volume of Charge:

Final Volume:

Volume %	:	IBP	10	20	30	40	50
°F	:						
Volume %	:	60	70	80	90		
°F	:						

Run No. A-54

Catalyst No. C-54

Catalyst Composition

Metals : 4%CoO 2%MoO₃ 1%NiO 2%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 428 SRC-II Feed Temperature, °C : 85

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .87

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 76

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

wt% N₂ : - .090 .010 .030 .310 .330 .350 .430

wt% S : - - - - .600 - - .340

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
O_F :

Volume % : 60 70 80 90
O_F :

Run No. A-55

Catalyst No. C-55

Catalyst Composition

Metals : 4%CoO 2%MoO₃ 4%NiO 8%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 420 SRC-II Feed Temperature, °C : 80

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .96

H₂: Oil Ratio, scf/bbl : 10,000

Yield of Oil, Volume % (balance is gas, coke & holdup)
: 58

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

wt% N₂ : - .030 .140 .350 .290 .320 .370 .500

wt% S : - - - - .625 - - .285

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

Run No. A-56

Catalyst No. C-56

Catalyst Composition

Metals : 4%CoO 2%MoO₃ 4%NiO 2%WO₃

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

Base Surface Area, m²/g : 214.57

Base Pore Volume, ml/g : .8397

Base Pore Diameter (4V/A), Å : 156.5

Base Median Pore Diameter, Å : 161

Run Temperature, °C : 420 SRC-II Feed Temperature, °C : 85

Run Pressure, psig : 1,000

Liquid Hourly Space Velocity : .96

H₂: Oil Ratio, scf/bbl : 10,000

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

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

wt% N₂ : - .060 .100 .240 .490 .010 .620 .840

wt% S : - - - - .720 - - .505

ASTM Distillation

Volume of Charge:

Final Volume:

Volume % : IBP 10 20 30 40 50
°F :

Volume % : 60 70 80 90
°F :

APPENDIX B

VARIABLE	1	2	3
MEAN (12)=	31.81	82.50	3.404
STD DEV =	12.46	26.76	.3308
SKENNESS =	1.724	.0000	.9868
KURTOSIS =	5.453	1.731	3.598
MAXIMUM =	65.40	120.0	4.181
MINIMUM =	18.80	45.00	2.934

DEPENDENT VARIABLE = 3
 INDEPENDENT VARIABLES = 2

FIT:	VAR	R-PART	B	SE(B)	T	F-VALUE
	2	-.8347	-.1032E-01	.2154E-02	-4.792	.0000

INTERCEPT = 4.256
 R-SQUARED = .6966

ANALYSIS OF VARIANCE:

SOURCE	DF	S. S.	M. S.	F-VALUE	P-VALUE
REGRESS	1	.8388	.8388	22.96	.0000
RESIDUAL	10	.3653	.3653E-01		
TOTAL	11	1.204			

VARIABLES DESCRIPTION:

1 = %DN for Nalco-78-6008C-1/32" base material

2 = running time, t, in minutes

3 = ln %DN

REGRESSION EQUATION: $\ln \%DN = 4.256 - 0.01 t$

APPENDIX C

VARIABLE	1 6	2 7	3 8	4 9	5 10
MEAN (112)=	71.81	92.14	3.000	5.000	2.500
	5.000	15.50	9611.	.1103E 07	.1356E 09
STD DEV =	18.25	33.62	1.004	3.013	1.507
	3.013	4.630	6608.	.1072E 07	.1658E 09
SKEWNESS =	-.2458	.2834	.0000	.0000	.0000
	.0000	.0000	.7696	1.162	1.451
KURTOSIS =	2.061	2.040	1.000	1.000	1.000
	1.000	2.256	2.518	3.164	3.762
MAXIMUM =	99.10	150.0	4.000	8.000	4.000
	8.000	24.00	.2250E 05	.3375E 07	.5062E 09
MINIMUM =	28.20	45.00	2.000	2.000	1.000
	2.000	7.000	2025.	.9113E 05	.4101E 07

DEPENDENT VARIABLE = 1
 INDEPENDENT VARIABLES= 2, 3, 10

FIT:	VAR	R-PART	B	SE(E)	T	F-VALUE
	2	-.7435	-.6365	.5509E-01	-11.55	.0000
	3	.4115	3.519	.7499	4.693	.0000
	10	.2981	.3625E-07	.1117E-07	3.245	.1574E-02

INTERCEPT = 115.0
 F-SQUARED = .8159

ANALYSIS OF VARIANCE:

SOURCE	DF	S.S.	M.S.	F-VALUE	P-VALUE
REGRESS	3	.3015E 05	.1005E 05	159.6	.0000
RESIDUAL	108	6802.	62.98		
TOTAL	111	.3695E 05			

VARIABLES DESCRIPTION:

- | | |
|---------------------------------|-------------------------------|
| 1 = %DN | 6 = wt% WO ₃ |
| 2 = running time, t, in minutes | 7 = summation of metal oxides |
| 3 = wt% CoO | 8 = t ² |
| 4 = wt% MoO ₃ | 9 = t ³ |
| 5 = wt% NiO | 10 = t ⁴ |

APPENDIX D

VARIABLE	1	2	3	4	5
	6	7			
MEAN (16)=	38.04	61.64	49.80	3.000	5.000
	2.500	5.000			
STD DEV =	26.10	10.93	16.49	1.033	3.098
	1.549	3.098			
SKENNESS =	.1411	-1.856	-.1684	.0000	.0000
	.0000	.0000			
KURTOSIS =	1.921	5.636	2.498	1.000	1.000
	1.000	1.000			
MAXIMUM =	84.70	70.10	77.40	4.000	8.000
	4.000	8.000			
MINIMUM =	.0000	29.90	14.90	2.000	2.000
	1.000	2.000			

DEPENDENT VARIABLE = 3
 INDEPENDENT VARIABLES= 4, 5, 7

FIT:	VAR	R-PART	B	SE(E)	T	P-VALUE
	4	-.7758	-8.525	2.002	-4.259	.1112E-02
	5	.8342	3.496	.6672	5.239	.0000
	7	.5791	1.642	.6672	2.460	.3002E-01

INTERCEPT = 49.69
 R-SQUARED = .8114

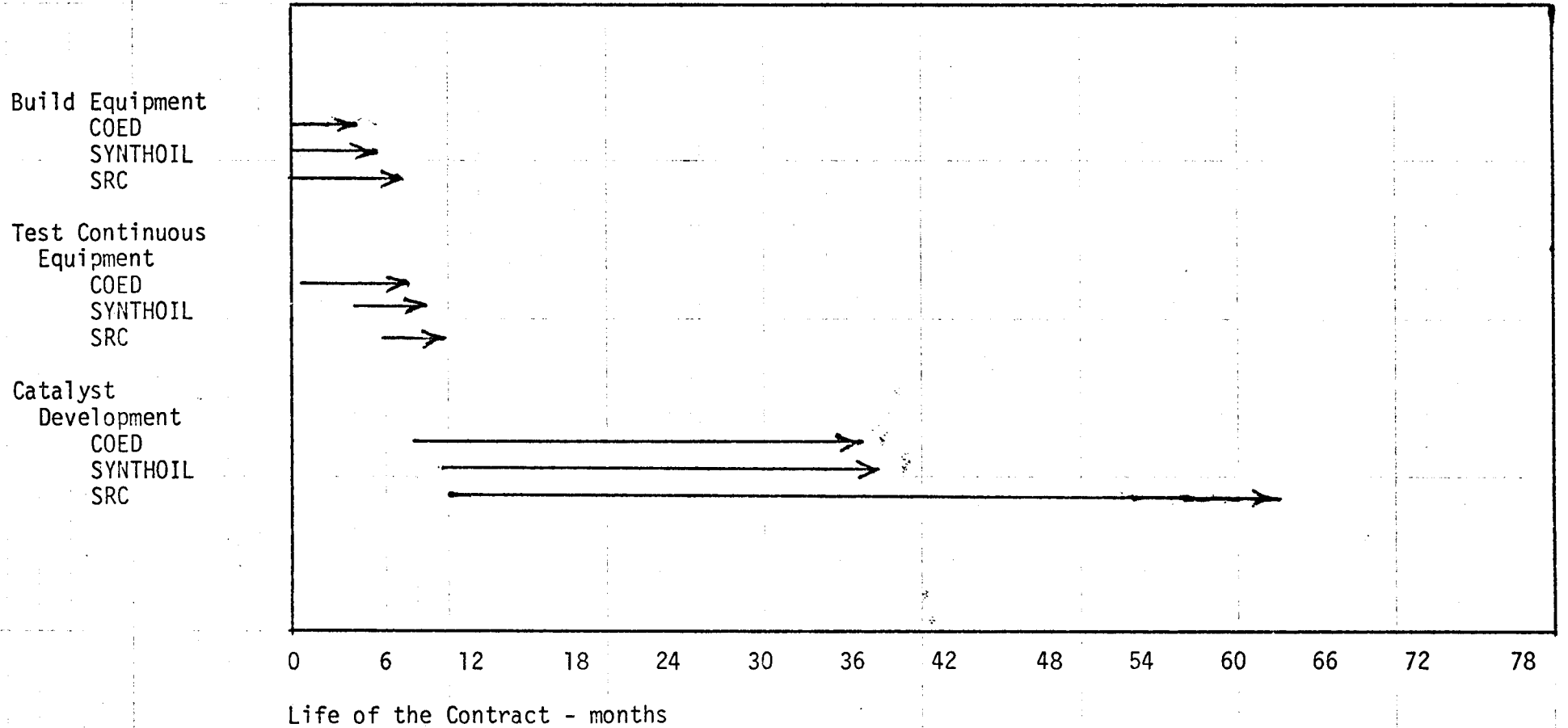
ANALYSIS OF VARIANCE:

SOURCE	DF	S. S.	M. S.	F-VALUE	P-VALUE
REFRESS	3	3311.	1104.	17.21	.0000
RESIDUAL	12	769.3	64.11		
TOTAL	15	4080.			

VARIABLES DESCRIPTION:

- 1 = %DS in 90 minutes
- 2 = %DS in 150 minutes
- 3 = average of variables 1 and 2
- 4 = wt% CoO
- 5 = wt% MoO₃
- 6 = wt% NiO
- 7 = wt% WO₃

PICTORIAL PROGRESS REPORT



Standard Form 1035 September 1973 Treasury Form 2000 1035-110		PUBLIC VOUCHER FOR PURCHASES AND SERVICES OTHER THAN PERSONAL CONTINUATION SHEET			VOUCHER NO. 36	
					SCHEDULE NO.	
					SHEET NO. 2	
U.S. DEPARTMENT, BUREAU, OR ESTABLISHMENT U.S. Energy Research and Development Admin.						
NUMBER AND DATE OF ORDER	DATE OF DELIVERY OR SERVICE	ARTICLES OR SERVICES <i>(Enter description, item number of contract or Federal supply schedule, and other information deemed necessary)</i>	QUAN- TITY	UNIT PRICE		AMOUNT
				COST	PER	
Montana State University Bozeman, Montana G&C 26000789	59717	Contract Number EX-76-C-01-2034 "Catalytic Hydrogenation of Coal Derived Liquids", Principal Investigator - Dr. Lloyd Berg				
For the period:		June 1, 1980 through June 30, 1980				
				Current period		Cumulative to date
Salaries & Wages				3,513	50	103,117.96
Benefits				367	28	6,018.42
Supplies & Materials				1,685	28	33,486.09
Travel						3,978.91
Equipment						12,441.92
Awards (Tuition & Fees)						5,755.30
Indirect Costs				2,231	45	60,875.29
		Sub Total...		7,797	51	225,673.89
Cost Sharing				1,376	03	38,015.72
		TOTAL...		9,173	54	263,689.61

SHOULD YOU HAVE ANY QUESTIONS
 CONCERNING FEDERAL MATTERS ON
 THIS REPORT, PLEASE CONTACT:
 THE I. KORN ADM. ASSOC.
 1000 WEST PINE STREET
 BOZEMAN, MONTANA 59715
 PHONE: 406-994-2381

Standard Form 1035
September 1975
Treasury Form 2000
1045-110

PUBLIC VOUCHER FOR PURCHASES AND SERVICES OTHER THAN PERSONAL

CONTINUATION SHEET

VOUCHER NO.
37
SCHEDULE NO.
SHEET NO.
2

U.S. DEPARTMENT, BUREAU, OR ESTABLISHMENT

U. S. Energy Research and Development Admin.

NUMBER AND DATE OF ORDER	DATE OF DELIVERY OR SERVICE	ARTICLES OR SERVICES <i>(Enter description, item number, contract or Federal supply schedule, and other information deemed necessary)</i>	QUANTITY	UNIT PRICE		AMOUNT
				COST	PER	
Montana State University Bozeman, Montana 59717 G&C 26000789				Contract No. EX-76-C-01-2034 "Catalytic Hydrogenation of Coal Derived Liquids", principal investigator - Dr. Lloyd Berg		
For the period:		July 1, 1980 through August 31, 1980				
				CURRENT PERIOD		CUMULATIVE TO DATE
				4,822	75	107,940.71
				591	77	6,610.19
				1,613	30	35,099.39
						3,978.91
				183	93	12,625.85
						5,755.30
				3,113	35	63,988.64
		Subtotal		10,325	10	235,998.99
		Cost Sharing		1,680	83	39,696.55
		Total		12,005	93	275,695.54

SHOULD YOU HAVE ANY QUESTIONS REGARDING FISCAL MATTERS ON THIS REPORT, PLEASE CONTACT: ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION, DEPARTMENT OF ENERGY, BOZEMAN, MONTANA 59715, PHONE: 406-994-2381

EXHIBIT 4
U.S. ATOMIC ENERGY COMMISSION

UNIVERSITY-TYPE CONTRACTOR'S RECOMMENDATION FOR
DISPOSITION OF SCIENTIFIC AND TECHNICAL DOCUMENT

(See Instructions on Reverse Side)

1. AEC REPORT NO. EX-76-C-01-2034-27	2. TITLE CATALYTIC HYDROGENATION OF COAL-DERIVED LIQUIDS.
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3. TYPE OF DOCUMENT (Check one):

a. Scientific and technical report

b. Conference paper:

Title of conference _____

Date of conference _____

Exact location of conference _____

Sponsoring organization _____

c. Other (Specify)

4. RECOMMENDED ANNOUNCEMENT AND DISTRIBUTION (Check one):

a. AEC's normal announcement and distribution procedures may be followed.

b. Make available only within AEC and to AEC contractors and other U.S. Government agencies and their contractors.

5. REASON FOR RECOMMENDED RESTRICTIONS:

6. SUBMITTED BY: NAME AND POSITION (Please print or type)

Lloyd Berg, Professor

Organization

Department of Chemical Engineering
Montana State University
Bozeman, MT. 59717

Signature 	Date September 18, 1980
--	----------------------------

FOR AEC USE ONLY

7. AEC CONTRACT ADMINISTRATOR'S COMMENTS, IF ANY, ON ABOVE ANNOUNCEMENT AND DISTRIBUTION RECOMMENDATION:

8. PATENT CLEARANCE:

a. AEC patent clearance has been granted by responsible AEC patent group.

b. Report has been sent to responsible AEC patent group for clearance.

c. Patent clearance not required.