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DEMETALLIZATION OF HEAVY RESIDUAL OILS - PHASE II

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DEMETALLIZATION OF HEAVY RESIDUAL OILS - PHASE II

by

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ABSTRACT

A new low cost demetallization catalyst for heavy residual oils was developed at the Trenton Laboratory of Hydrocarbon Research, Inc., a subsidiary of Dynalectron Corporation, during Phase I of Contract No. 68-02-0293, funded by the Environmental Protection Agency. The purpose of Phase II was to optimize the level of promoter metal on the support, produce a batch on commercial scale, and evaluate the commercially produced catalyst by demetallizing vacuum residua containing different levels of contaminant metals. Screening runs were conducted on laboratory samples of activated bauxite impregnated with low levels of molybdenum which were prepared by Minerals and Chemicals Division of Engelhard Corporation. To check out commercial production capabilities, a commercial production run was made on the best support-promoter combination as determined from the screening runs. The commercial production catalyst was tested for activity and aging characteristics and results were compared to the best laboratory prepared catalyst.

Two vacuum residuum feedstocks were demetallized, the products desulfurized over high activity commercial HDS catalyst beads, and costs to produce low sulfur fuel oil were calculated and compared against costs using unpromoted activated bauxite.

Descriptions of test units, operating conditions, and procedures are given, including detailed run summaries, as well as tables presenting feedstock, product, and catalyst inspections.

Graphs and tables depicting operating costs for producing 0.5 weight percent sulfur fuel oil are given, along with projected costs for producing 0.3 and 1.0 weight percent sulfur fuel oil. Conclusions based on experimental results are also discussed.

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GLOSSARY

MM	Millions
1 Angstrom (Å)	10^{-8} Centimeters
g/cc	Grams/cubic centimeter
M^2/g	Square meters/gram
Mesh Sizes	Mesh sizes are all United States Standard Sieve Series
psig	Pounds per square inch, gauge
SCF/Bbl	Standard cubic feet of gas per barrel of oil (60°F, 1 Atm.)
L.S.V.	Liquid Space Velocity, Volume of Oil/Hour/ Volume of Reactor
$V_o/Hr/V_r$	Volumes of Oil/Hour/Volume of Reactor
C.S.V.	Catalyst Space Velocity, Barrels of Oil/Day/ Pound of Catalyst
Bbl/D/Lb	Barrels of Oil/Day/Pound of Catalyst
BPSD	Barrels per Stream Day
ppm	Parts per million
SFS	Saybolt Furol Seconds
SUS	Saybolt Universal Seconds
V.B.	Vacuum Bottoms = Vacuum Residuum

SECTION I

CONCLUSIONS

Granular 20 x 50 mesh activated bauxite impregnated with 1.0 weight percent molybdenum proved to be better than a 0.5 weight percent molybdenum and equal to a 2.0 weight percent molybdenum preparation in terms of demetallization and long life when used to demetallize Tia Juana vacuum residuum under the standard testing conditions.

Commercial technology and facilities are presently available to at least one major catalyst manufacturer to produce this catalyst on a commercial scale; the catalyst being equivalent in all respects to the best laboratory prepared plant simulation sample.

Process economics for production of low sulfur fuel oil were obtained by utilizing the commercial production catalyst in a demetallization stage followed by a desulfurization stage over commercial high activity HDS catalyst beads. High metals Tia Juana vacuum residuum and low metals Gach Saran vacuum residuum were processed and costs calculated. To produce 0.5 weight percent sulfur fuel oil in a United States Gulf Coast plant of 20,000 barrels per stream day capacity, including capital charges of 25 percent of investment, from Tia Juana vacuum residuum, the operating cost per barrel was $1.63 versus $1.69 for unpromoted activated bauxite and investment cost of $17.07 MM for promoted commercial catalyst versus $19.44 MM for unpromoted activated bauxite. From Gach Saran vacuum residuum, the comparable costs were $1.41 per barrel versus $1.47 and $16.06 MM versus $17.22 MM.

SECTION II

RECOMMENDATIONS

In order to more accurately define technical operating parameters, further demonstrate flexibility of the process, and obtain more accurate operating cost figures, the following program is recommended:

1. On a third feedstock, other than Tia Juana or Gach Saran, demetallize over commercial production demetallization catalyst to three different levels of metals removal. For example, 45 to 50 percent vanadium removal, 65 to 70 percent vanadium removal, and 80 to 85 percent vanadium removal.

2. Desulfurize low level metals removal product from item (1) above over high activity HDS catalyst beads to produce low sulfur fuel oil product.

3. Using medium level metals removal product from item (1) above, desulfurize at three different conditions over HDS beads.

4. Desulfurize high level metals removal product from item (1) above over HDS beads.

5. Using a fourth different feedstock, demetallize the vacuum residuum over commercial production demetallization catalyst to two different levels of vanadium removal. For example, 45 to 50 percent vanadium removal, and 80 to 85 percent vanadium removal.

6. Desulfurize lower level metals removal product from item (5) above over HDS beads.

7. Desulfurize higher level metals removal product from item (5) above over HDS beads at different operating conditions.

Incorporating data from the above work with data obtained from previous work on this project, technical and economic evaluations would be made to more closely define operating conditions and costs for a process to produce low sulfur (0.3 to 1.0 W %) fuel oil. Upon the successful completion of the above tasks, a detailed commercial plant design based on these results should be made.

SECTION III

INTRODUCTION

In Phase I work of the Environmental Protection Agency Contract No. 68-02-0293 on demetallization of heavy residual oils, the goals were to develop a low cost demetallization catalyst at laboratory scale level, to test the catalyst in pilot plant operations, and to make a preliminary economic evaluation for producing low sulfur fuel oil from heavy residual oils using the newly developed catalyst system in a demetallization stage, followed by a desulfurization stage using commercial HDS beads.

These goals were achieved by the development of a 20 x 50 mesh activated bauxite impregnated with small (0.5 to 2.0 W %) amounts of molybdenum and the successful testing of this catalyst in a demetallization stage of a two-stage demetallization-desulfurization operation to produce low sulfur fuel oil from Tia Juana, Bachaquero, and Gach Saran vacuum residua.

Economic evaluations showed this catalyst to offer substantial cost advantages over unpromoted bauxite when used in a demetallization stage of a two-stage system. The scope of the work under Phase II was to optimize promoter level on the 20 x 50 mesh activated bauxite support, to explore commercial technology for producing the catalyst, and to evaluate commercially produced catalyst in order to further define costs of producing low sulfur fuel oil in a two-stage demetallization-desulfurization process.

Long term aging tests on laboratory prepared catalysts containing 0.5, 1.0, and 2.0 weight percent molybdenum promoter showed the 1.0 weight percent molybdenum catalyst to be superior to the 0.5 weight percent molybdenum catalyst with respect to demetallization activity and aging and about equal to the 2.0 weight percent molybdenum catalyst with respect to demetallization activity while showing slightly better aging characteristics. On this basis, Minerals and Chemicals Division of Engelhard Corporation was contracted to produce a 10,000 pound batch

of 20 x 50 mesh activated bauxite impregnated with 1.0 weight percent molybdenum using commercial production equipment. This commercially prepared catalyst proved to be equal in all respects to the best laboratory prepared plant simulation catalyst sample.

Three vacuum residua were demetallized over the commercially produced catalyst and the demetallized products from two feeds were desulfurized over commercial HDS beads. From these data, costs for producing low sulfur fuel oil were calculated and compared against costs when using unpromoted activated bauxite in a demetallization stage.

SECTION IV

EXPERIMENTAL PROGRAM

UNIT DESCRIPTION

All runs were carried out in continuous, downflow, fixed bed reactor systems. A schematic diagram is shown in Figure 1. The reactor, fabricated of one-and-a-half inch O.D. by one-inch I.D. stainless steel tubing, has a catalyst bed length of approximately 16 inches. A drawing of the reactor tube is shown in Figure 2. The volume (loose) of catalyst charged to the reactor was 200 cc. Provision was made for an internal thermocouple which is positioned in the center of the catalyst bed approximately midway between the top and bottom. Heat to the reactor was supplied by a lead bath.

The melted charge stock was pumped to reactor pressure with a metering pump, mixed with hydrogen makeup gas, and fed to the top of the reactor. The hydrogen concentration of the makeup gas was 100 percent and no recycle of the exit gases was employed. In the reactor, the feed was contacted with the catalyst. The mixed vapor and liquid product from the reactor was cooled and passed to a high pressure receiver from which gas was sampled, metered, and vented. The net product was let down in pressure and passed to a low pressure receiver from which gas was sampled periodically, metered, and vented. The liquid product was collected and weighed periodically. Upon completion of a run, the catalyst was removed from the reactor for inspection and/or analyses. Three essentially identical units, 184, 185, and 201, were used for these runs.

A standard procedure was devised to screen the demetallization catalysts in short term operations. This consisted of an initial startup period which conditioned the fresh catalyst at lower temperatures for a short period of time. This startup schedule was as follows:

Figure 1. FIXED BED DEMETALLIZATION UNIT

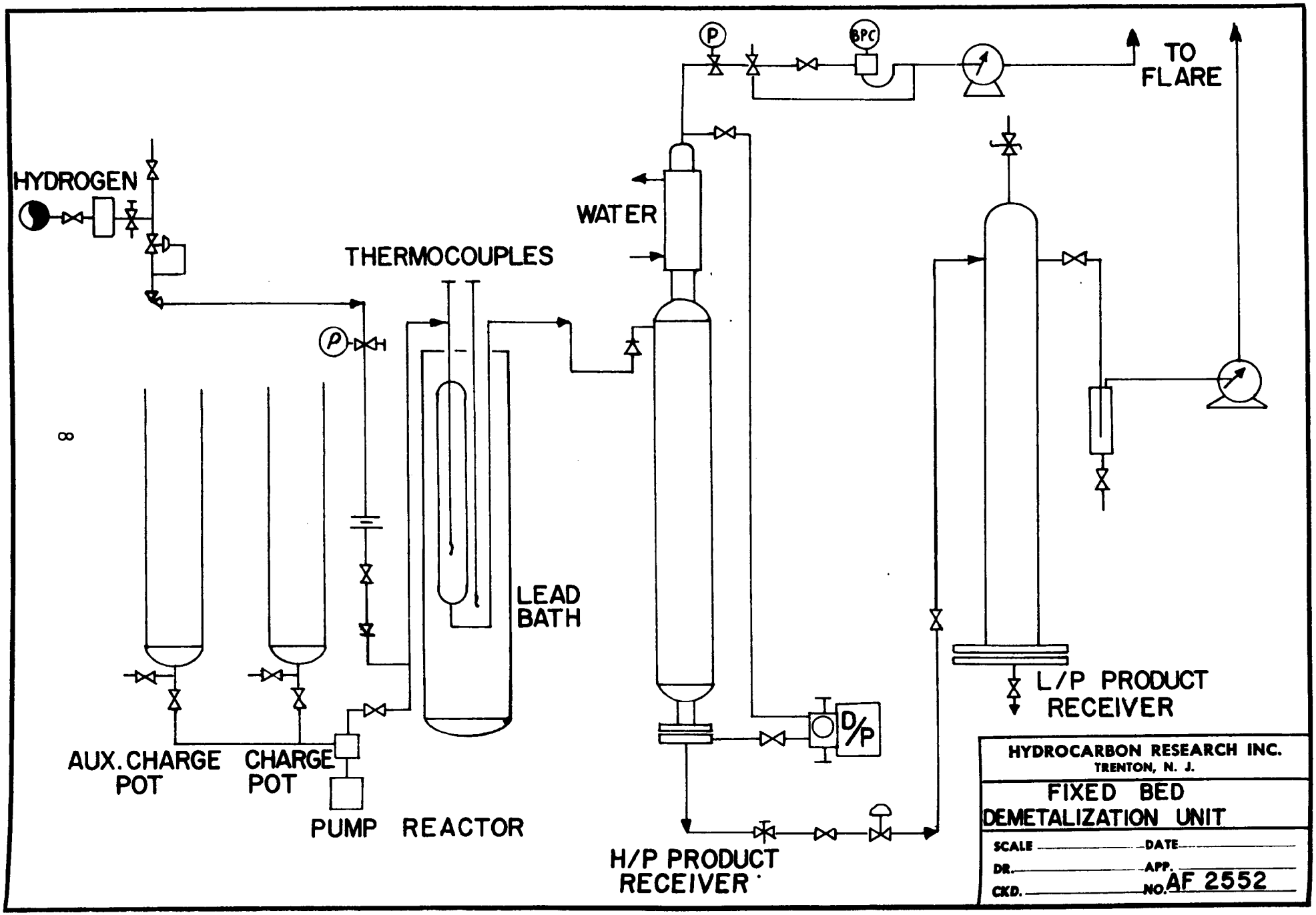
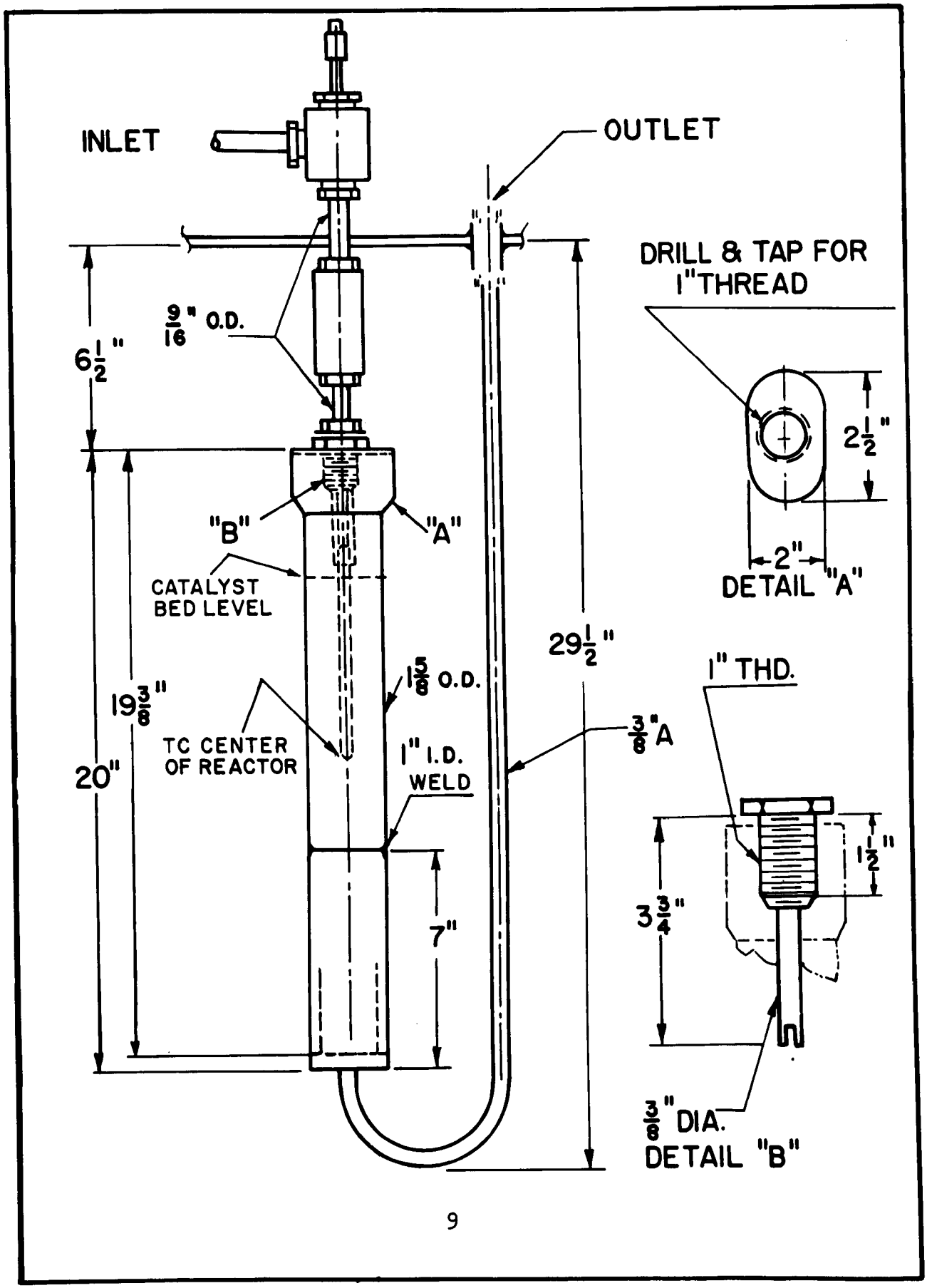


Figure 2. FIXED BED DEMETALLIZATION REACTOR



Period	------- 1A -------			1B, 2, Etc.
Temperature, °F	750	775	790	790
Pressure, psig	2000	2000	2000	2000
Hydrogen Rate, SCF/Bbl	4000	4000	4000	4000
Liquid Space Velocity, $V_O/Hr/V_r$	0.75	0.75	0.75	0.50
Time on Temp., Hrs.	4	4	1	Continue at above conditions until shutdown.

After the unit was lined out at 790°F, the period was ended and the remainder of the run continued at 790°F, 2000 psig, 4000 SCF/Bbl, and 0.50 $V_O/Hr/Vr$ for a period of two to fifteen days, depending upon the performance of the catalyst being screened.

In Phase I work, simple first order kinetics was used to describe the rate of vanadium removal using unpromoted as well as promoted activated bauxite. However, preliminary studies, over the range of space velocities used in the screening runs, indicated that pseudo first order kinetics more closely describe the rate of vanadium removal over activated bauxite promoted with 2.0 weight percent molybdenum. The kinetic equation used to correct for variations in space velocities and obtain rate constants for use later in this program is given in equation (1):

$$K_M = (C.S.V.)^{0.5} \times \ln \frac{V_F}{V_P} \qquad (1)$$

where C.S.V. = Catalyst Space Velocity, Bbl Oil/Day/Lb Cat.
V_F = Vanadium in Feed, in ppm
V_P = Vanadium in Product, in ppm

FEED SELECTION AND PREPARATION

Three vacuum residuum feeds, Tia Juana, Bachaquero, and Gach Saran, were used for all demetallization runs.

Tia Juana and Bachaquero crudes originate in the Lake Maracaibo area of Venezuela. In 1973, the total production of Tia Juana

and Bachaquero crudes was 120 and 216 million barrels, equivalent to 36 and 104 million barrels of vacuum residual oils, with estimated crude reserves of about 1,702 and 2,041 million barrels, respectively. Tia Juana vacuum residuum and Bachaquero crude were obtained from the Creole Petroleum Corporation, a subsidiary of Exxon. The Bachaquero vacuum residuum used in this work was prepared at the HRI® Laboratory by distillation of the atmospheric residuum obtained from Bachaquero crude. Gach Saran crude originates in the Persian/Arabian Gulf in Iran. In 1973, the production rate of Gach Saran crude was 324 million barrels, equivalent to 75 million barrels of vacuum residual oil, with an estimated crude reserve of about 8,140 million barrels. The vacuum residuum feed was obtained from Kashima Oil Company of Japan. The three feeds are representative of major high metals crudes available in the world and are, for the most part, sold as export material. Detailed inspections of the three vacuum residua are presented in Table 1.

The Gach Saran vacuum residuum obtained from Kashima Oil Company has a slightly higher sulfur than those given in the literature. This is due to the presence of a small percentage of Kuwait Vacuum Bottoms in the feed obtained from Kashima.

DEMETALLIZATION CATALYST

Table 2 summarizes inspections on the 20 x 50 mesh demetallization catalyst obtained from Minerals and Chemicals Division of Engelhard Corporation as part of the 10,000 pound production using commercial manufacturing facilities. For the runs carried out, about 184 grams of this catalyst with a static volume of 200 cc was charged to the reactor. Pore size distribution on this catalyst is given later in Figure 10.

CHOOSING A CATALYST FOR COMMERCIAL PRODUCTION

In our previous work under Phase I of the present project, 20 x 50 mesh activated bauxite, promoted with low levels of molybdenum (0.5 to 2.0 W % Mo), proved to be the most effective catalyst of those tested for removing contaminant metals (vanadium and nickel) from heavy residua.

Table 1. INSPECTIONS ON VACUUM RESIDUUM FEEDSTOCKS

Feedstock	Tia Juana Vacuum Residuum	Gach Saran Vacuum Residuum	Bachaquero Vacuum Residuum
HRI Identification No.	3615	3574	L-388
Gravity, °API	7.8	6.9	5.3
Sulfur, W %	2.9	3.72	3.49
Ramsbottom Carbon, W %	17.8	18.0	20.4
Carbon, W %	84.98	86.30	85.16
Hydrogen, W %	10.59	10.48	10.24
H/C Atomic Ratio	1.48	1.45	1.43
Nitrogen, ppm	5800	6850	7400
Vanadium, ppm	570	291	754
Nickel, ppm	75	110	96
Viscosity, SFS @ 210°F	1126	1310	
IBP-975°F, V %	12.0	6.0	8.0
Gravity, °API	17.3	17.7	16.3
Sulfur, W %	2.11	2.54	2.85
975°F+, V %	88.0	84.0	92.0
Gravity, °API	6.5	6.1	4.7
Sulfur, W %	3.18	3.81	3.66
Ramsbottom Carbon, W %	21.2	19.2	22.7

Table 2. DESCRIPTION AND ANALYSES OF THE 1% MOLYBDENUM IMPREGNATED BAUXITE

HRI Identification Number	3634
Size	20 x 50 U.S. Mesh
Molybdenum, W %	1.06
Volatile Matter, W %	2.0
Bulk Density, g/cc	1.01
Pore Volume, cc/gm	0.282
Screen Analyses, W %	
20/30 Mesh	52.4
30/40 Mesh	30.7
40/50 Mesh	16.9

Initial activity of the 0.5 weight percent molybdenum was only slightly lower than the 2.0 weight percent molybdenum. However, there was some doubt as to whether the 0.5 weight percent molybdenum catalyst would sustain its activity as well as the 2.0 weight percent molybdenum. Since the difference in molybdenum content between 0.5 weight percent and 2.0 weight percent catalyst represents about $0.03 or more per pound of molybdenum, it was evident that a more extensive evaluation of lower promoter level catalyst was necessary.

LONG TERM DEACTIVATION STUDY ON 0.5 WEIGHT PERCENT MOLYBDENUM CATALYST

A laboratory prepared sample of 20 x 50 mesh activated bauxite impregnated with 0.5 weight percent molybdenum was made and designated as HRI LX-28. Demetallization of Tia Juana vacuum bottoms under standard operating conditions was carried out in Run 184-182 over LX-28. Unfortunately, it was found that it was necessary to run at 0.5 volume of oil per hour per volume of reactor ($V_O/Hr/V_r$) to achieve the same level of demetallization as was obtained at 0.75 $V_O/Hr/V_r$ using the 2.0 weight percent molybdenum catalyst. In addition, the desulfurization level achieved was lower using the lower promoted catalyst.

Figure 3 and Figure 4 compare the demetallization and desulfurization results of this run with earlier experimental work (Run 184-174) on the 2.0 weight percent molybdenum laboratory prepared catalyst, LX-22.

ECONOMIC COMPARISON OF 0.5 WEIGHT PERCENT MOLYBDENUM CATALYST VERSUS 2.0 WEIGHT PERCENT MOLYBDENUM CATALYST

To compare the economics of the two catalysts, it was necessary to determine the overall cost of the demetallization and desulfurization steps. Demetallization catalyst LX-28 (0.5 W % Mo) is less active than LX-22 (2.0 W % Mo), both in demetallization and desulfurization. Results obtained in Phase I of this contract indicated that the second order rate constant

$$K = (B/D/Lb) \times (S_F/S_P-1)$$

Figure 3. DEMETALLIZATION AGING RUNS OVER 20 X 50 MESH BAUXITE IMPREGNATED WITH 0.5 AND 2.0 W % MOLYBDENUM

Feed: Tia Juana Vacuum Residuum
Feed Composition: 7.5-7.7°API, 2.85-2.91 W % S, 541-586 ppm V, 71-74 ppm Ni

Legend	Run No.	Catalyst HRI No.	% Mo	Hydrogen Pressure psig	Temp. °F	$V_O/Hr/V_r$
○	184-182	LX-28	0.5	2000	790	0.5
△	184-174	LX-22	2.0	2000	790	0.75

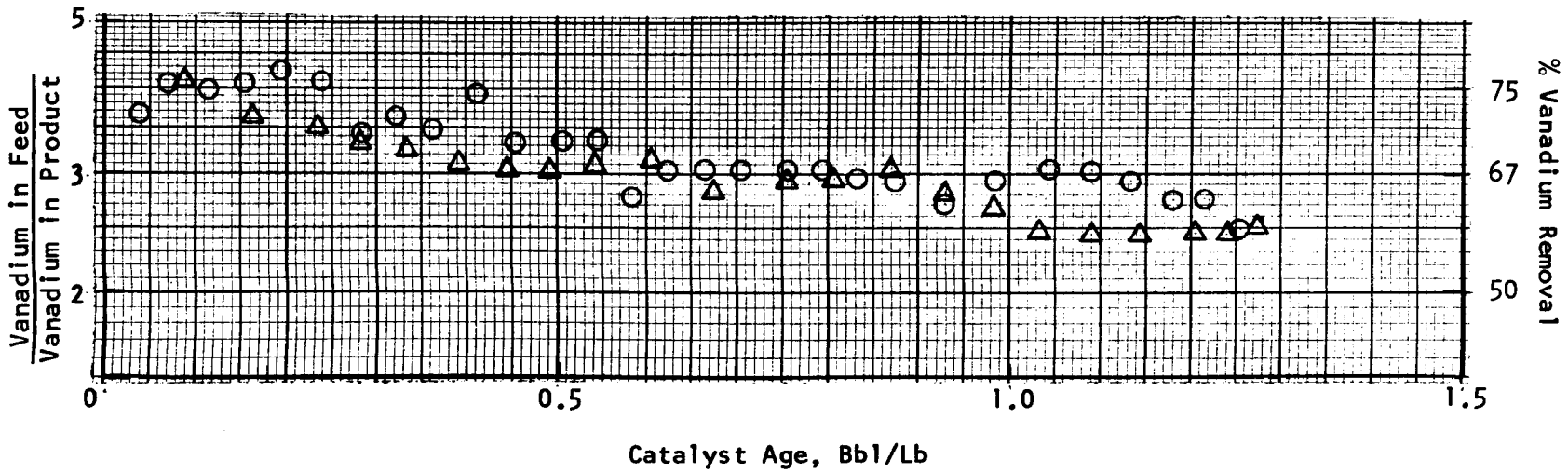
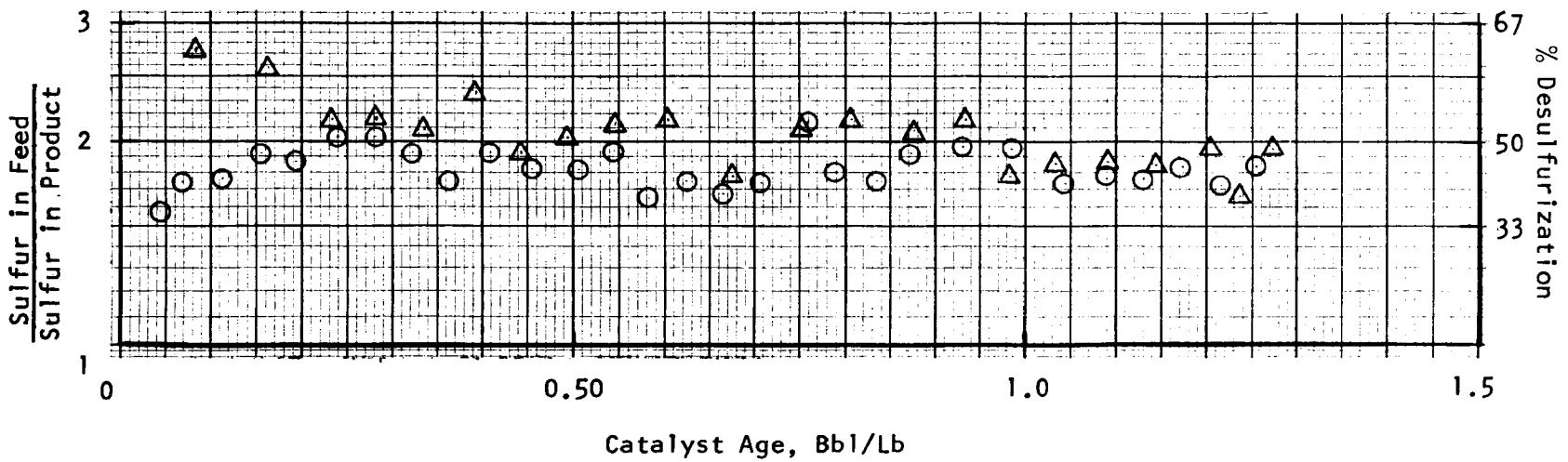


Figure 4. DESULFURIZATION OBTAINED DURING DEMETALLIZATION AGING RUNS OVER 20 X 50 MESH BAUXITE IMPREGNATED WITH 0.5 AND 2.0 W % MOLYBDENUM

Feed: Tia Juana Vacuum Residuum
Feed Composition: 7.5-7.7°API, 2.85-2.91 W % S, 541-586 ppm V, 71-74 ppm Ni

Legend	Run No.	Catalyst HRI No.	% Mo	Hydrogen Pressure psig	Temp. °F	$V_o/Hr/V_r$
○	184-182	LX-28	0.5	2000	790	0.5
△	184-174	LX-22	2.0	2000	790	0.75



in the desulfurization stage decreases with increase in sulfur removal in the demetallization stage. In order to obtain the initial rate constant for estimating the desulfurization operating conditions for producing the required final product, an eight-day desulfurization run (201-83) was conducted over the HDS beads using the feed demetallized over LX-28. As expected, the desulfurization rate constant was slightly higher for the higher sulfur feed prepared with LX-28. To produce fuel oil containing the same level of sulfur, it was necessary, in spite of the higher rate constant, to operate the desulfurization stage for this feed at more severe conditions than those used for the feed prepared with LX-22 because of the higher level of sulfur in the demetallized feed from the LX-28 operation. This resulted in higher overall operating costs when compared with the case using LX-22. The cost difference between the LX-28 and LX-22 catalysts should be about $0.13 per pound for the two catalysts to be equivalent. This amount of difference is unlikely since the difference in raw chemical costs between the two promoter levels is only about $0.03 per pound.

The results from the evaluation of the 0.5 weight percent molybdenum catalyst indicated the optimum level of promoter metal must be somewhat higher than 0.5 weight percent.

EVALUATION OF CATALYST SAMPLES PREPARED BY MINERALS AND CHEMICALS DIVISION OF ENGELHARD CORPORATION

In order to minimize the cost of this catalyst, it is necessary to keep the manufacturing process as simple as possible. Arrangements were made with Minerals and Chemicals Division of Engelhard Corporation to prepare samples in their laboratory which they felt could be produced by them at minimum cost. Minerals and Chemicals is the producer of Porocel, the activated bauxite chosen as the support for this catalyst.

Initially, they prepared a sample by impregnating 20 x 50 mesh activated bauxite with 1.0 weight percent molybdenum using HRI's technique to assure that comparable results could be achieved by both laboratories. The next seven samples, all containing 2.0 weight percent molybdenum, represented successively easier methods of preparation. The final three samples, all representing the easiest plant simulation preparation technique, contained 2.0, 1.0, and 0.5 weight percent molybdenum.

All catalyst samples prepared by Minerals and Chemicals were subjected to our standard short term screening test using standard operating conditions on Tia Juana vacuum residuum. Table 3 summarizes the results of these screening tests. Catalyst LX-22, prepared by HRI, is included as the reference catalyst. Minerals and Chemicals sample No. 1 (HRI 3581), laboratory method, not a plant production simulation method, was superior with respect to metals removal and equal in desulfurization to LX-22. The next seven samples, Minerals and Chemicals No. 3, 6, 7, 8, 9, 10, and 11, were all about equal to each other and to LX-22. The 0.5 weight percent molybdenum preparation, Minerals and Chemicals sample No. 13 (HRI 3609), was definitely inferior to all other samples including LX-22.

Sample No. 14 (HRI 3610), representing the easiest plant simulation preparation method, and Sample No. 12 (HRI 3608), using the same easiest preparation method, but containing 1.0 weight percent molybdenum, showed 79 percent vanadium removal compared to 77 percent for LX-22. Both samples had higher nickel removal rates than LX-22 and only in desulfurization did the 1.0 weight percent molybdenum catalyst fall below LX-22.

On the basis of results from all screening runs, the last 2.0 weight percent molybdenum sample, Minerals and Chemicals No. 14 (HRI 3610) and the 1.0 weight percent molybdenum sample, Minerals and Chemicals sample No. 12 (HRI 3608) were chosen for further aging tests.

AGING TEST ON THE TWO BEST MINERALS AND CHEMICALS LABORATORY-PREPARED PLANT SIMULATION CATALYSTS

Aging tests were performed in our standard test units using Tia Juana vacuum residuum and standard testing conditions. The 2.0 weight percent molybdenum catalyst (HRI 3610) was tested in Run 184-186 lasting 22 days and the 1.0 weight percent molybdenum catalyst (HRI 3608) was tested in Run 185-231 lasting 26 days. Figure 5 shows the plots of demetallization activity (vanadium removal) versus catalyst age in barrels of oil per pound of catalyst (Bbl/Lb) for the two catalysts being tested, as well as a plot of an HRI laboratory-prepared 2.0 weight percent molybdenum catalyst (LX-22-5) previously run in 184-174 to serve as a base for comparison.

Table 3. EVALUATION OF MINERALS & CHEMICALS SAMPLES

DEMETALLIZATION OF TIA JUANA VACUUM RESIDUUM

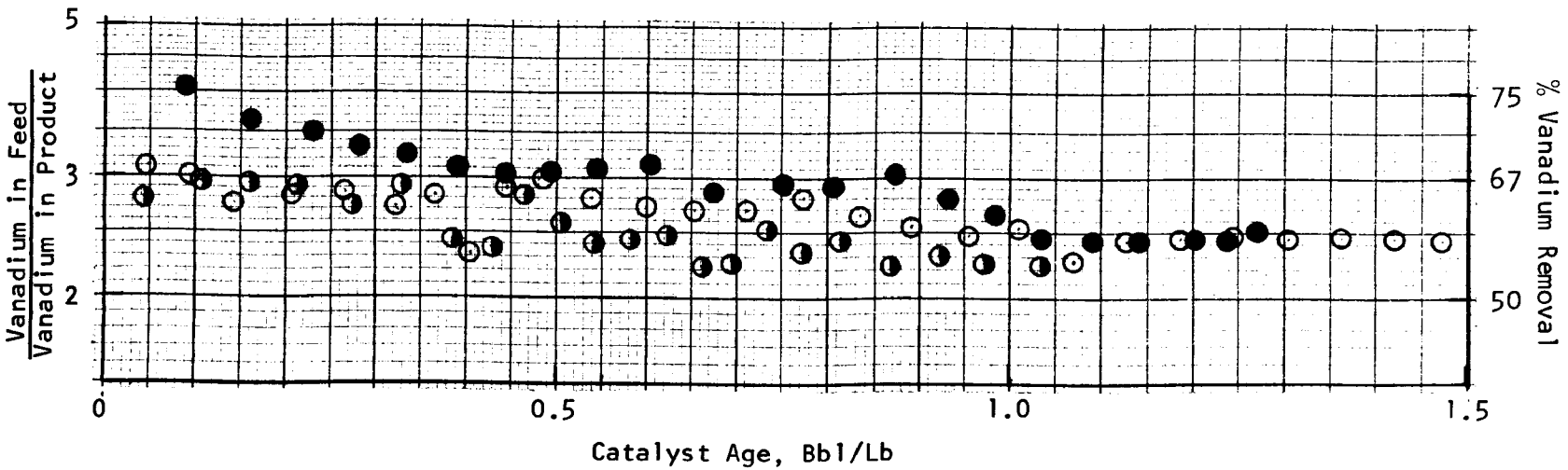
Catalyst, Bbl/Lb	0.10
Temperature, °F	790
Hydrogen Pressure, psig	2000
Liquid Space Velocity, $V_o/Hr/V_r$	0.5

Catalyst	HRI Number	% Mo	Evaluation Run	Product Concentrations			% Removal		
				%S	ppm V	ppm Ni	S	V	Ni
LX-22		2.0	184-166	1.00	127	45	65	77	39
M & C 3231-7, Sample #1	3581	2.0	185-224	1.00	85	35	65	84	52
M & C 3231-4, Sample #6	3582	2.0	185-225	1.58	134	39	45	75	47
M & C 3231-11, Sample #7	3583	2.0	185-226	1.04	133	49	64	75	34
M & C Sample #10	3598	2.0	185-227	1.05	125	38	63	78	49
M & C Sample #11	3599	2.0	184-183	1.12	127	39	61	77	47
M & C Sample #3	3594	2.0	185-228	1.00	118	39	65	79	47
M & C Sample #8	3596	2.0	184-184	1.00	128	42	65	77	43
M & C Sample #9	3597	2.0	185-229	1.04	122	41	64	78	45
M & C Sample #12	3608	1.0	184-185	1.20	109	39	60	79	44
M & C Sample #13	3609	0.5	185-230	1.49	154	44	50	70	37
M & C Sample #14	3610	2.0	201-84	1.02	106	34	66	79	51

Figure 5. DEMETALLIZATION AGING RUNS OVER 20 X 50 MESH BAUXITE IMPREGNATED WITH 1.0 AND 2.0 W % MOLYBDENUM

Feed: Tia Juana Vacuum Residuum
Feed Composition: 7.7-8.0°API, 2.85-2.98 W % S, 520-585 ppm V, 70-74 ppm Ni

Legend	Run No.	Catalyst HRI No.	% Mo	Hydrogen Pressure psig	Temp. °F	Actual $V_o/Hr/V_r$	Data Corrected to $V_o/Hr/V_r$
●	184-174	LX-22-5	2.0	2000	790	0.36-0.99	0.50
○	185-231	3608	1.0	2000	790	0.50-0.75	0.50
◑	184-186	3610	2.0	2000	790	0.50-0.75	0.50



Up to a catalyst age of 0.5 Bbl/Lb, the activity of the two catalysts is about equal, but below the LX-22-5 catalyst. There is no apparent explanation for the unusually high initial demetallization activity exhibited by LX-22-5. From age 0.5 Bbl/Lb to the end of the run, the 1.0 weight percent molybdenum catalyst exhibited slightly higher demetallization rates over the 2.0 weight percent molybdenum catalyst and had about the same rate as the LX-22-5 catalyst at the end of the run.

From this technical evaluation, neither catalyst exhibited clear cut superior qualities over the other. However, the 1.0 weight percent molybdenum catalyst, having about $0.02 per pound cost advantage because of the lower molybdenum loading, was chosen for the commercial production run.

SHORT TERM TESTING OF COMMERCIALLY PREPARED 1.0 WEIGHT PERCENT MOLYBDENUM CATALYST

Minerals and Chemicals Division of Engelhard Corporation was contracted to produce 10,000 pounds of 1.0 weight percent molybdenum on 20 x 50 mesh activated bauxite using their commercial manufacturing facilities. In their opinion, this amount of catalyst would provide enough on stream time to provide a representative sample of catalyst.

The production catalyst (HRI 3634) was subjected to our standard five-day screening test in Run 184-189, along with a sample representing a variation in the commercial production (HRI 3635) in Run 185-233. The demetallization results of these two catalyst samples are plotted in Figure 6, along with a plot of previously run Minerals and Chemicals sample containing 1.0 weight percent molybdenum (HRI 3608) in Run 184-185 for comparison. Figure 7 represents the desulfurization results on these catalysts from the same runs.

The variation in production catalyst (HRI 3635) showed no significant difference with respect to demetallization and desulfurization when compared to the regular production catalyst (HRI 3634).

Figure 6. DEMETALLIZATION SCREENING RUNS

OVER 20 X 50 MESH BAUXITE IMPREGNATED WITH 1.0 W % MOLYBDENUM

Feed: Tia Juana Vacuum Residuum
Feed Composition: 7.7-8.0°API, 2.85-2.98 W % S, 520-585 ppm V, 70-74 ppm Ni

Legend	Run No.	Catalyst HRI No.	% Molybdenum	Hydrogen Pressure psig	Temperature °F	LSV V_o/Hr/Vr	CSV B/D/Lb
○	184-185	3608	1	2000	790	0.5	0.039
◑	185-233	3635	1	2000	790	0.5	0.034
●	184-189	3634	1	2000	790	0.5	0.038

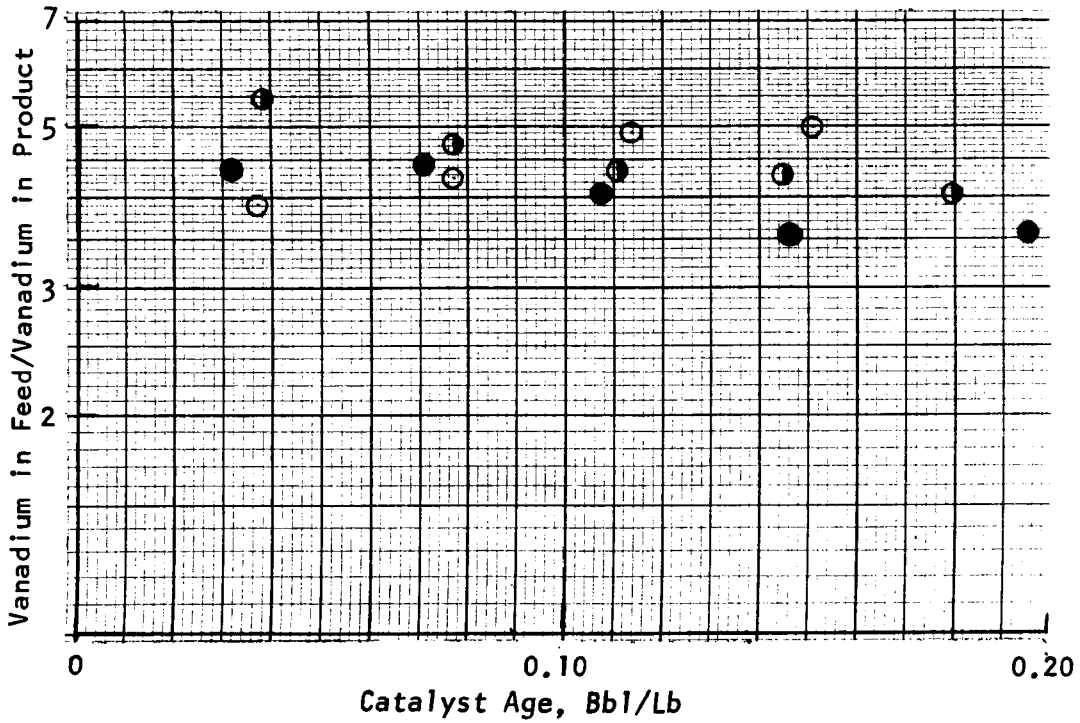
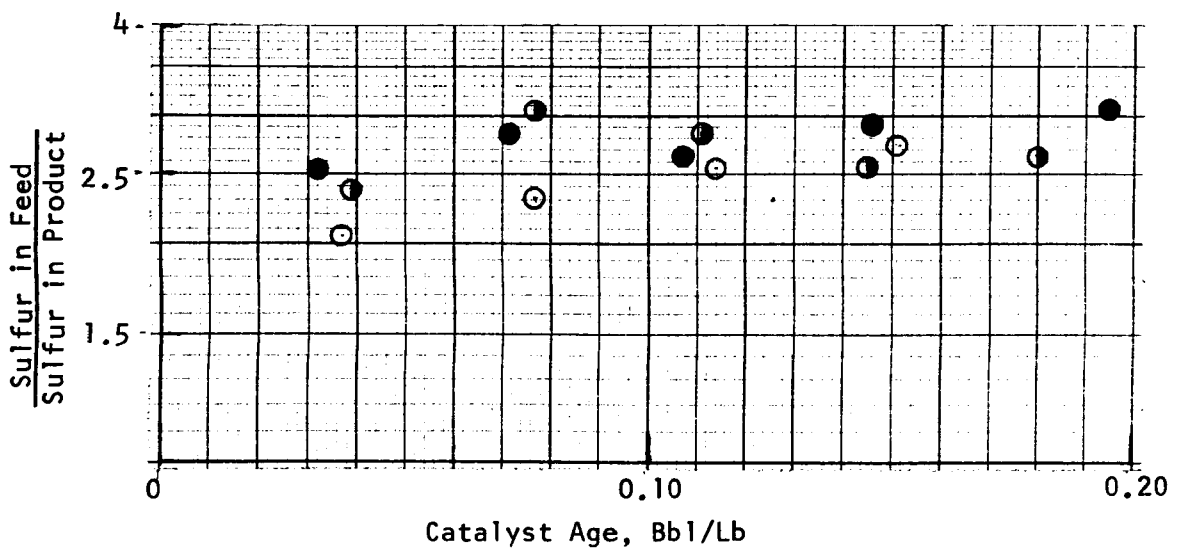


Figure 7. DESULFURIZATION OBTAINED DURING DEMETALLIZATION SCREENING RUNS

OVER 20 X 50 MESH BAUXITE IMPREGNATED WITH 1.0 W % MOLYBDENUM

Feed: Tia Juana Vacuum Residuum
Feed Composition: 7.7-8.0°API, 2.85-2.98 W % S, 520-585 ppm V, 70-74 ppm Ni

Legend	Run No.	Catalyst HRI No.	% Molybdenum	Hydrogen Pressure, psig	Temperature °F	LSV $V_o/Hr/V_r$	CSV B/D/Lb
○	184-185	3608	1	2000	790	0.5	0.039
◑	185-233	3635	1	2000	790	0.5	0.034
●	184-189	3634	1	2000	790	0.5	0.038



On the basis of the five-day screening test, the commercial production catalyst was accepted to be equal to the laboratory-prepared plant simulation 1.0 weight percent molybdenum catalyst with respect to demetallization and desulfurization. To test deactivation rates, aging tests were scheduled using Tia Juana vacuum residuum as well as other feedstocks.

Detailed operating conditions and liquid product inspections for each screening run of this series are given in Appendix A.

SECTION V

DEMETALLIZATION RUNS

DEMETALLIZATION OPERATING CONDITIONS

Demetallization operating conditions were selected so that a modest catalyst deactivation was maintained during the run. These conditions were chosen based on the results obtained in the Phase I work of this contract. All demetallization runs were carried out at a hydrogen pressure of 2000 psig, a reactor temperature of 790°F, a liquid space velocity of 0.75 V_O/Hr/V_R, and a hydrogen rate of about 4000 to 4500 SCF/B of feed. The runs were generally carried out to a catalyst age of about 1.3 to 1.6 Bbl/Lb. Runs of this duration provide a good measure of the catalyst deactivation rate which can be translated to the catalyst utilization rate for a given level of vanadium removal. Detailed operating conditions and liquid product inspections for each run of this series are given in Appendix B.

LONG TERM DEMETALLIZATION OF TIA JUANA VACUUM RESIDUUM

A long term run (184-190) of 24 days duration was conducted on Tia Juana vacuum residuum over commercially prepared demetallization catalyst (1.0 W % molybdenum on 20 x 50 mesh bauxite). The purpose of this run was to study the aging rate of this catalyst and also to produce feed for a desulfurization study. The operating conditions and the demetallization and desulfurization results are given in Figures 8 and 9.

Initial vanadium removal was about 69 percent. This value dropped to about 52 percent at a catalyst age of 1.35 Bbl/Lb. Nickel removal ranged from about 40 percent to about 24 percent, and sulfur removal was between 35 and 50 percent.

In the screening run (184-185) using HRI 3608, the vanadium removal was reported as 79 percent, which is higher than the

Figure 8 . DEMETALLIZATION OF TIA JUANA VACUUM RESIDUUM

OVER 1.0 W % MOLYBDENUM/20 X 50 MESH BAUXLTE

Run 184-190, Catalyst HRI No. 3634

Feed Composition	
Gravity, °API	7.8
Sulfur, W %	2.9
Vanadium, ppm	575
Nickel, ppm	75

Operating Conditions	
Hydrogen Pressure, psig	2000
Temperature, °F	790
Liquid Space Velocity, $V_F/Hr/V_R$	0.75
Catalyst Space Velocity, B/D/Lb	0.056
Hydrogen Rate, SCF/B (Vent)	4150

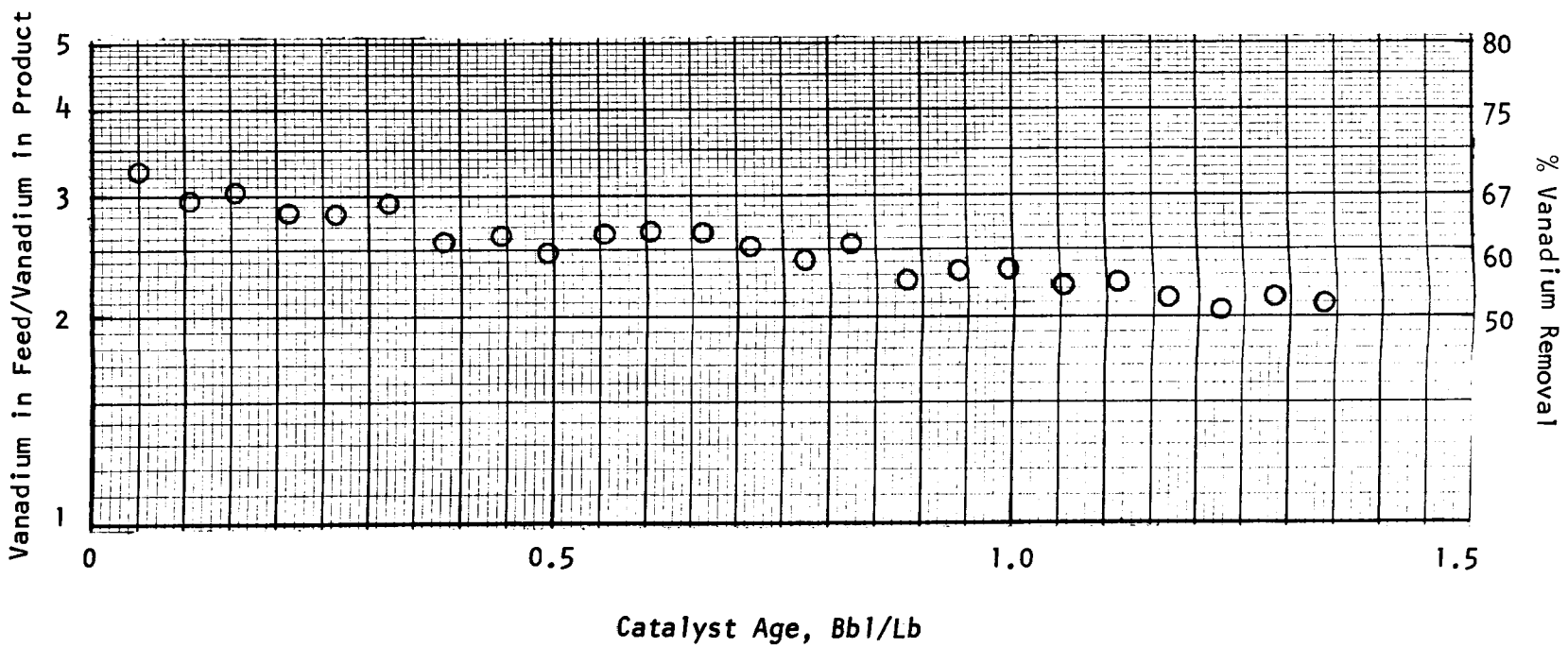


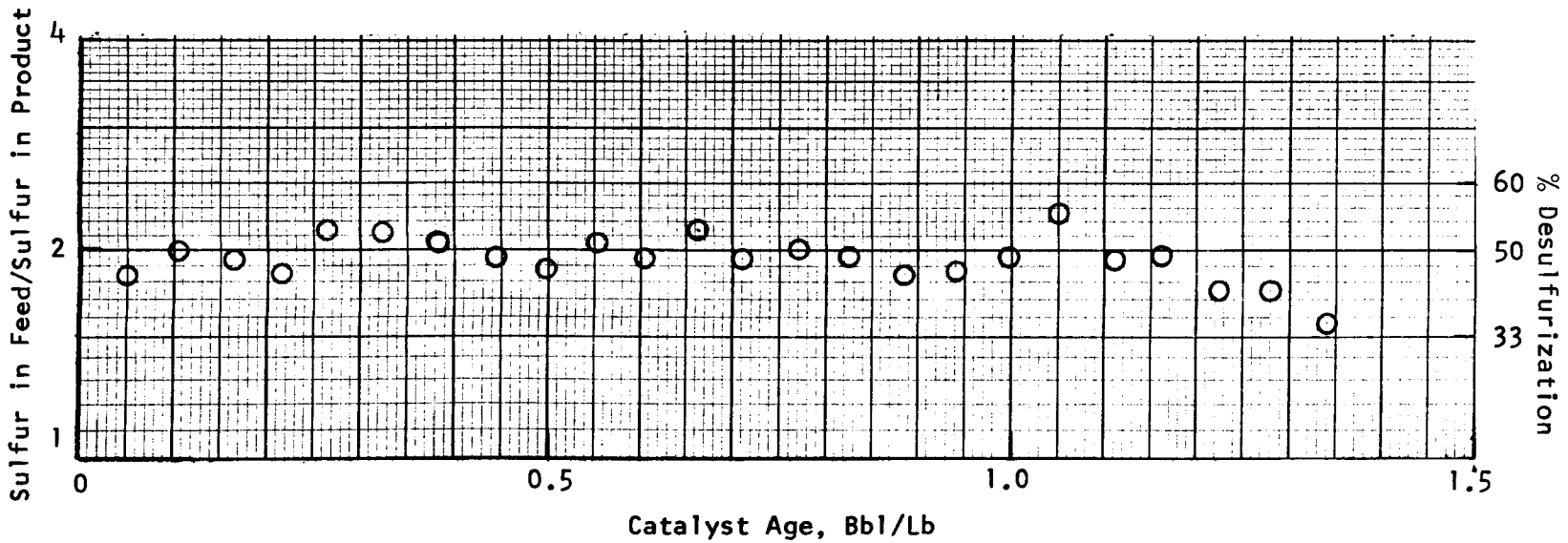
Figure 9. DESULFURIZATION OBTAINED DURING DEMETALLIZATION

OF TIA JUANA VACUUM RESIDUUM OVER 1.0 W % MOLYBDENUM/20 X 50 MESH BAUXITE

Run 184-190, Catalyst HRI No. 3634

Feed Composition	
Gravity, °API	7.8
Sulfur, W %	2.9
Vanadium, ppm	575
Nickel, ppm	75

Operating Conditions	
Hydrogen Pressure, psig	2000
Temperature, °F	790
Liquid Space Velocity, $V_F/Hr/V_R$	0.75
Catalyst Space Velocity, B/D/Lb	0.056
Hydrogen Rate, SCF/B (Vent)	4150



level obtained in this run. This is so because the screening run was conducted at a liquid space velocity of 0.5 $V_o/Hr/V_r$ whereas a space velocity of 0.75 $V_o/Hr/V_r$ was used in this run.

Figure 10 compares the difference in pore structure between the fresh and spent catalyst from Run 184-190, conducted with Tia Juana vacuum residuum. The pore volume of the spent catalyst was corrected to fresh basis using the following relationship:

$$\text{cc/gm Fresh Catalyst} = \frac{1}{1.000 - \Sigma Fi + \frac{1}{2}Fs} \times \text{cc/gm Spent Catalyst}$$

where Fi = weight fraction impurities on spent catalyst and
Fs = weight fraction sulfur on spent catalyst.

There is a small change in the pore volume in pores larger than 1000 angstroms (Å), while a substantial reduction is noted in pores between 1000 Å and 50 Å. All of the pore volume in pores less than 50 Å appears to be gone.

The results of the analysis of the spent catalyst from this operation, as well as from other demetallization operations, are presented in Table 4. They show a vanadium loading of about 12 percent and a carbon loading of about nine percent.

The variation of the demetallization rate constant with vanadium loading on the catalyst is given later in Figure 13.

LONG TERM DEMETALLIZATION OF GACH SARAN VACUUM RESIDUUM

Long term demetallization of Gach Saran vacuum residuum over commercially prepared demetallization catalyst (1.0 W % molybdenum on 20 x 50 mesh bauxite) was carried out in Run 185-235. The operating conditions and desulfurization and demetallization results of that operation are summarized in Figures 11 and 12. The purpose of this run was to investigate the aging characteristics of the catalyst and to produce feed for a desulfurization study. A high level of vanadium removal was accomplished with this feed. The level of vanadium removal and the rate of deactivation obtained with this catalyst were about the same as those obtained with the

Figure 10. CHANGE IN PORE SIZE DISTRIBUTION OF THE DEMETALLIZATION CATALYST WHEN DEMETALLIZING TIA JUANA VACUUM RESIDUUM

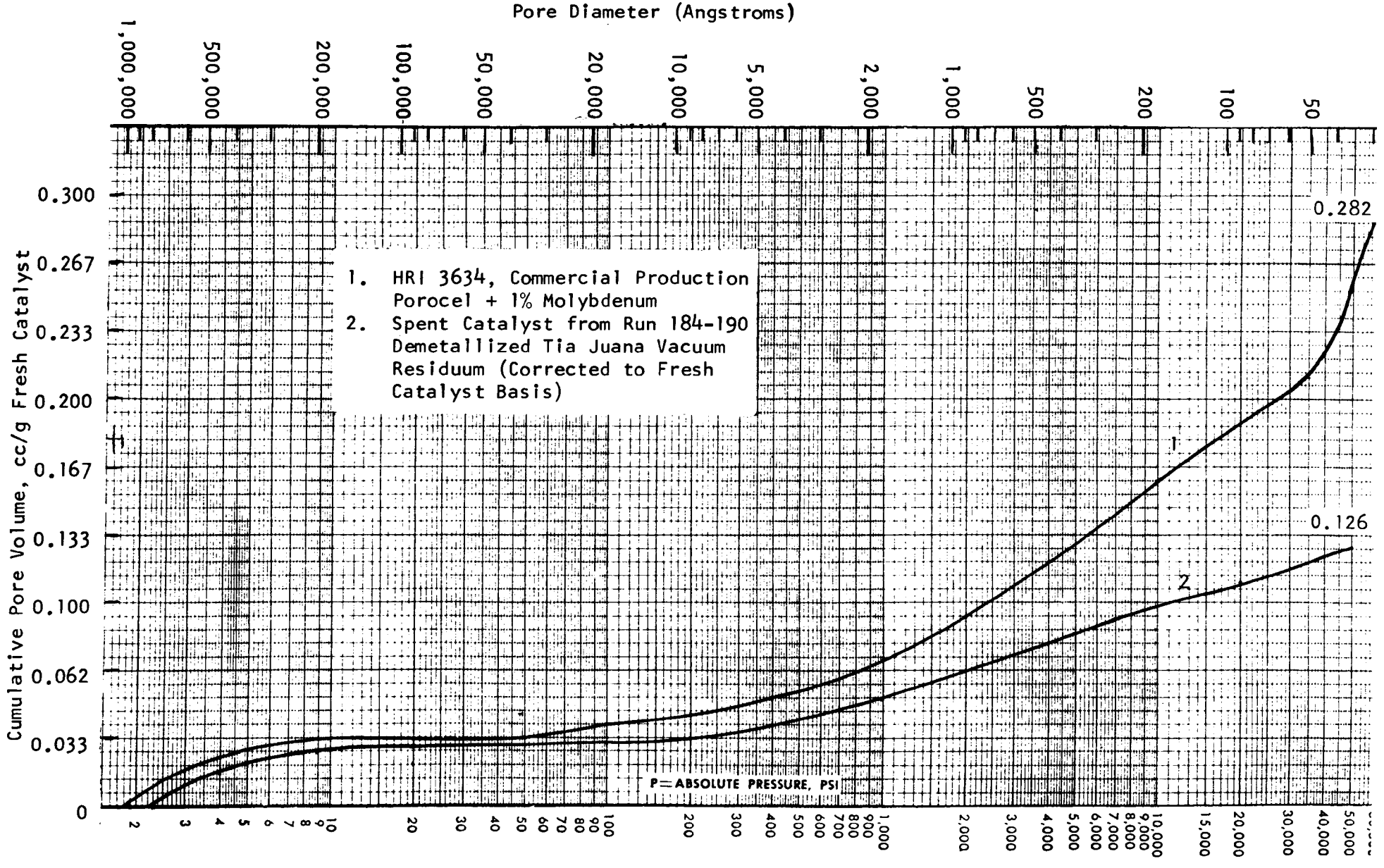


Table 4 . ANALYSES OF SPENT DEMETALLIZATION CATALYST

Run No.	Feed	Catalyst HRI No.	% Molybdenum	Weight Percent Element on Spent Catalyst			
				C	S	V	Ni
184-182	Tia Juana	LX-28	0.5	10.63	6.16	11.84	0.85
184-186	Tia Juana	3610	2.0	9.39	6.88	9.47	0.74
184-190	Tia Juana	3634	1.0	9.04	5.73	11.79	0.88
185-231	Tia Juana	3608	1.0	8.85	8.26	13.48	0.98
185-235	Gach Saran	3634	1.0	6.93	8.19	11.20	2.66
185-236	Bachaquero	3634	1.0	9.79	3.66	4.84	0.53

Figure 11. DEMETALLIZATION OF GACH SARAN VACUUM RESIDUUM

OVER 1.0 W % MOLYBDENUM/20 X 50 MESH BAUXITE

Run 185-235, Catalyst HRI No. 3634

Feed Composition	
Gravity, °API	6.9
Sulfur, W %	3.72
Vanadium, ppm	291
Nickel, ppm	110

Operating Conditions	
Hydrogen Pressure, psig	2000
Temperature, °F	790
Liquid Space Velocity, $V_F/Hr/V_R$	0.75
Catalyst Space Velocity, B/D/Lb	0.057
Hydrogen Rate, SCF/B (Vent)	4700

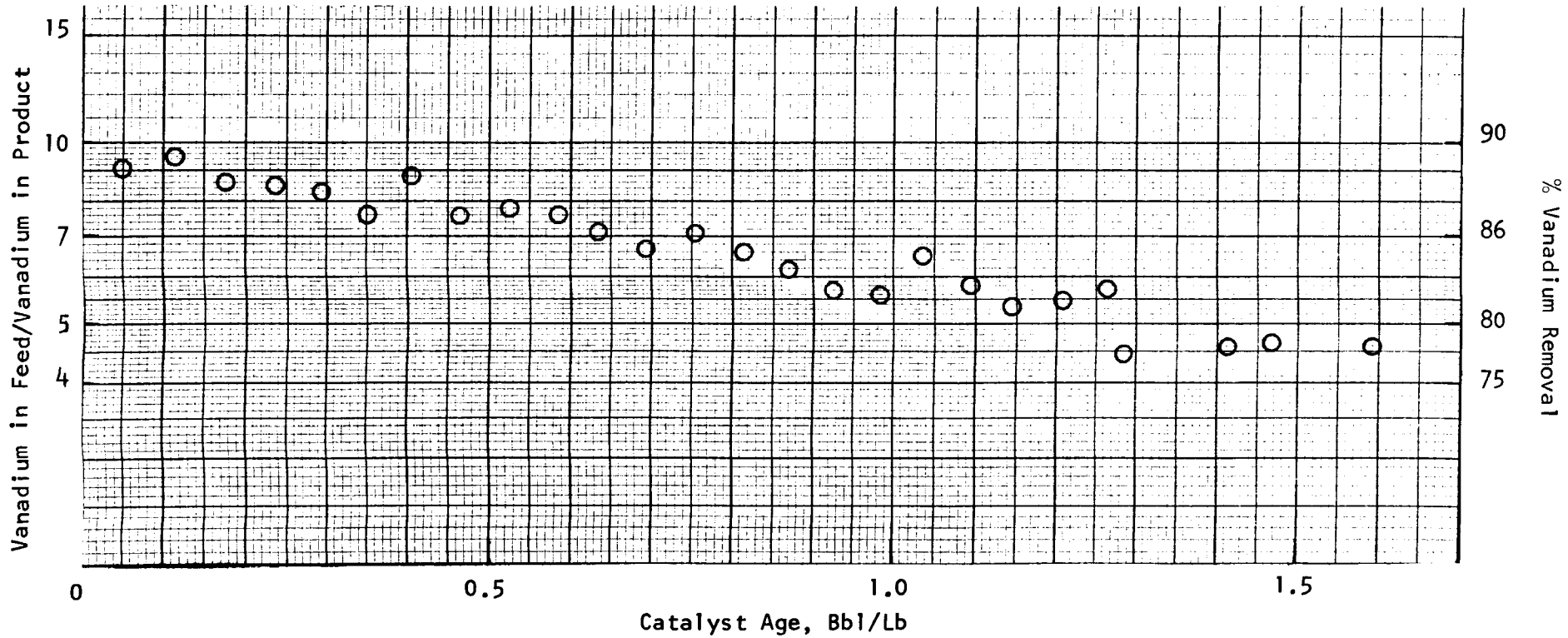


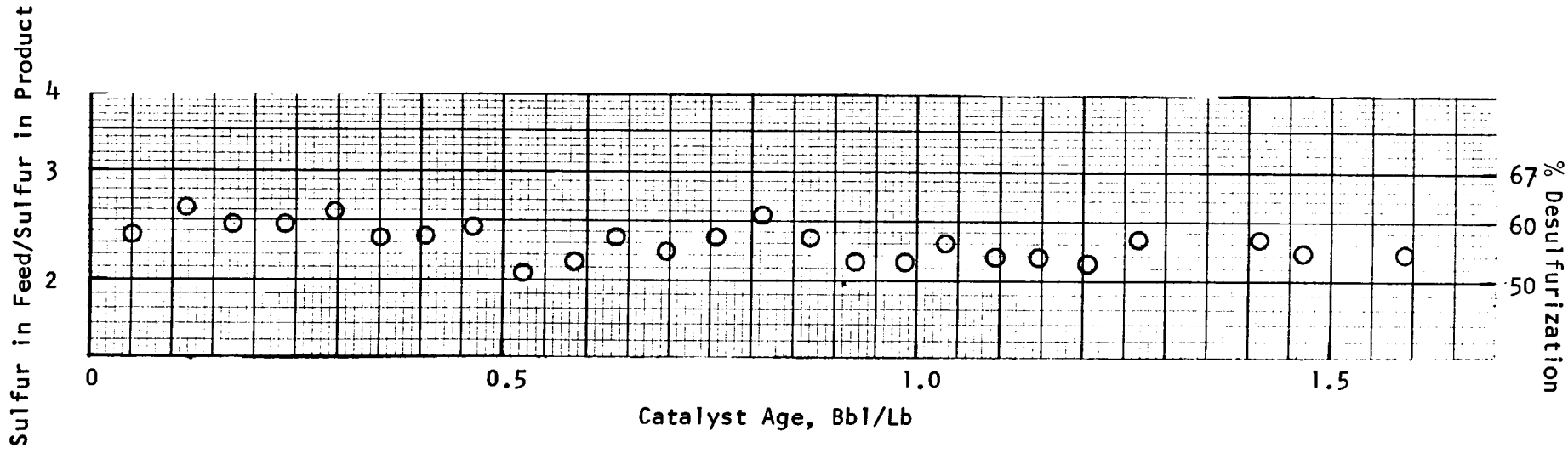
Figure 12. DESULFURIZATION OBTAINED DURING DEMETALLIZATION

OF GACH SARAN VACUUM RESIDUUM OVER 1.0 W % MOLYBDENUM/20 X 50 MESH BAUXITE

Run 185-235, Catalyst HRI No. 3634

Feed Composition	
Gravity, °API	6.9
Sulfur, W %	3.72
Vanadium, ppm	291
Nickel, ppm	110

Operating Conditions	
Hydrogen Pressure, psig	2000
Temperature, °F	790
Liquid Space Velocity, $V_F/Hr/V_R$	0.75
Catalyst Space Velocity, B/D/Lb	0.057
Hydrogen Rate, SCF/B (Vent)	4700



LX-22 catalyst (2.0 W % molybdenum on 20 x 50 mesh bauxite, prepared by HRI). Initial vanadium removal was about 89 percent and this value dropped to about 77 percent at a catalyst age of 1.6 Bbl/Lb. Nickel removal ranged from 63 to 75 percent and sulfur removal was about 55 percent. While the level of vanadium removal obtained with this feed was high, the rate of activity decline was modest when compared with the results of other feeds.

Figure 13 shows the variation of the demetallization rate constant as a function of vanadium loading on the catalyst for Gach Saran, Tia Juana, and Bachaquero feeds. For the same vanadium loading on the catalyst, the demetallization rate constant obtained with Gach Saran is about twice that obtained with Tia Juana.

In order to explain the large difference in demetallization rate constants between Tia Juana and Gach Saran residua, further work in characterizing these two residua may be of value. Perhaps the distribution of metals associated with the asphaltene and oil fractions is different for the two residua. Characterizing molecular size and species may also be helpful to explain the difference.

The reduction in pore volume of the catalyst that occurred during the process is shown in Figure 14. As is the case with the Tia Juana feed, a substantial reduction in pore volume in pores between 1000 Å and 50 Å is noted.

The results of the analysis of the spent catalyst from this run were presented previously in Table 4. They show a vanadium loading of 11.2 percent and a carbon loading of about 6.9 percent. This was the lowest value obtained when compared to the carbon loading from other runs.

DEMETALLIZATION OF BACHAQUERO VACUUM RESIDUUM

A short term run (185-236) of about five days was conducted on Bachaquero vacuum residuum over commercially prepared demetallization catalyst. The original objective of this run was to

Figure 13. VARIATION OF DEMETALLIZATION RATE CONSTANT WITH VANADIUM LOADING ON THE CATALYST

Feed

1. Gach Saran Vacuum Residuum
2. Bachaquero Vacuum Residuum
3. Tia Juana Vacuum Residuum

Operating Conditions

Hydrogen Pressure, psig	2000
Temperature, °F	790

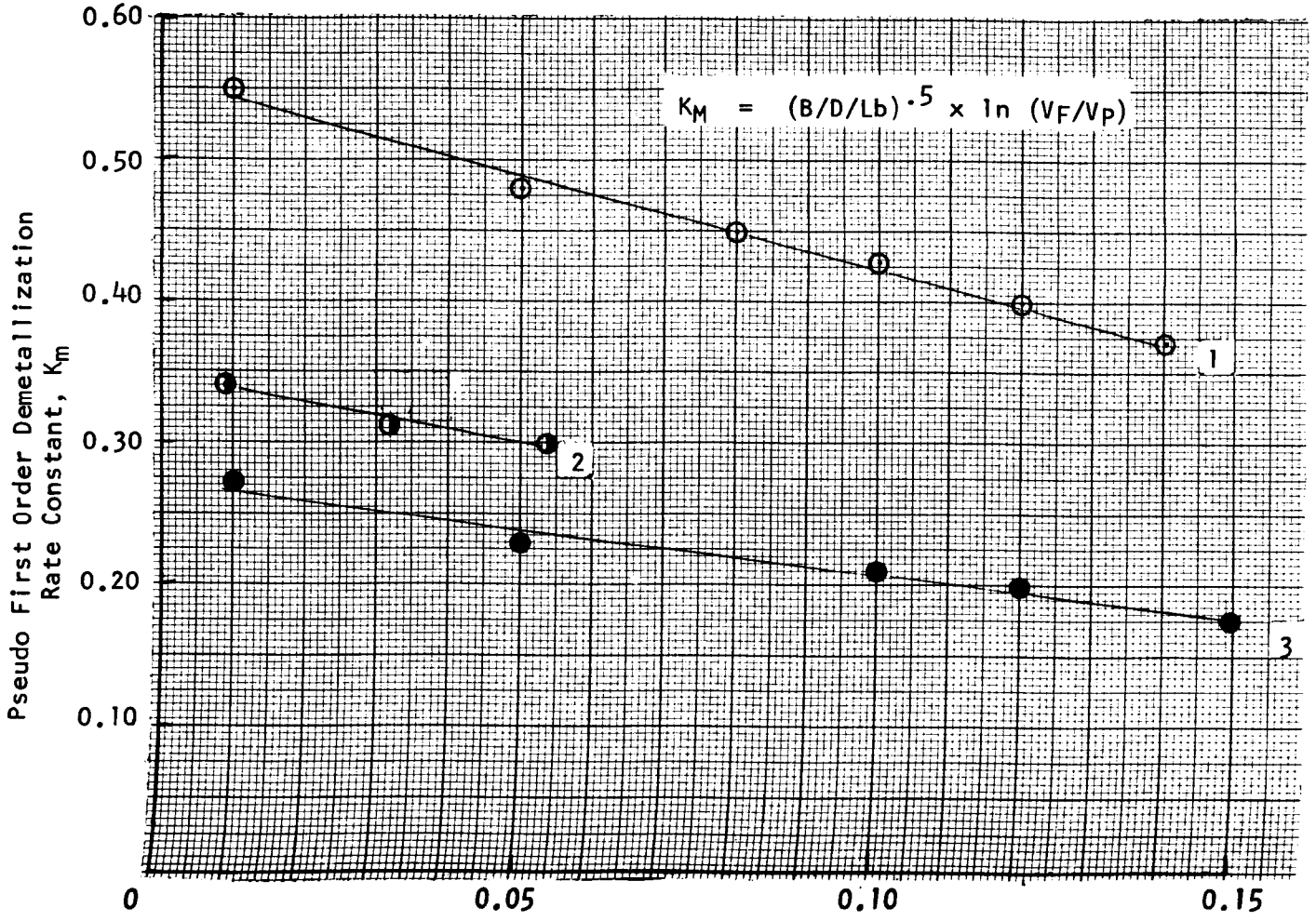
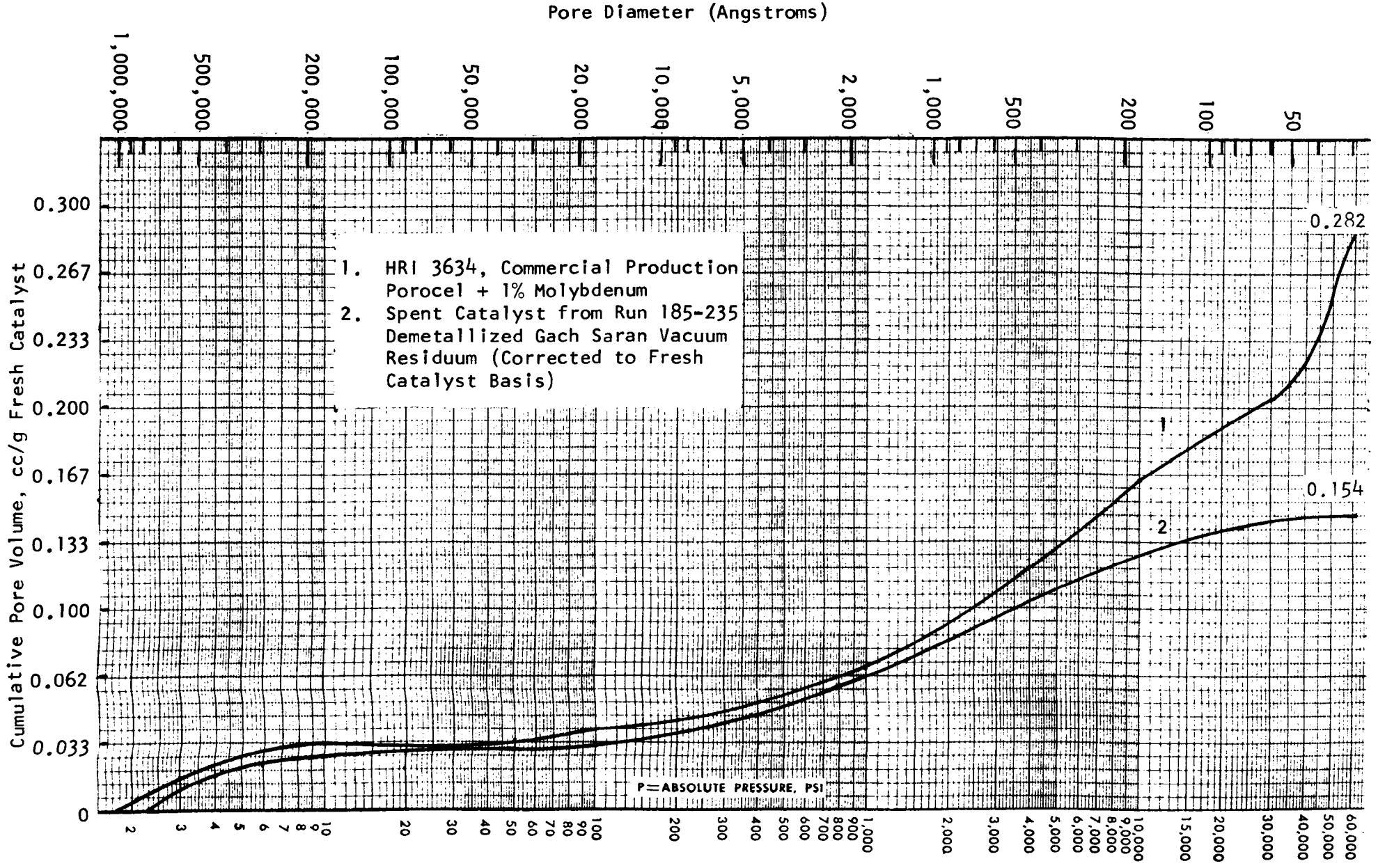


Figure 14. CHANGE IN PORE SIZE DISTRIBUTION OF THE DEMETALLIZATION CATALYST WHEN DEMETALLIZING GACH SARAN VACUUM RESIDUUM



study the aging characteristics of this catalyst when used with this feed and also to produce feed for a desulfurization study; however, due to lack of funds, the run had to be terminated. Operating conditions, demetallization and desulfurization results from this work are summarized in Figure 15.

The variation of the demetallization rate constant with vanadium loading on the catalyst was given previously in Figure 13. The rate constant obtained with this feed is slightly higher than that obtained with Tia Juana feed.

INSPECTIONS OF PRODUCTS FROM THE DEMETALLIZATION OPERATION

Detailed product inspections were obtained on one product from each of the demetallization operations on Tia Juana and Gach Saran vacuum residua over the commercially prepared 1.0 weight percent molybdenum catalyst. These inspections are given in Tables D-1 and D-2 in Appendix D. Chemical hydrogen consumption ranged from 350 SCF/B for Tia Juana vacuum residuum to 515 SCF/B for Gach Saran vacuum residuum.

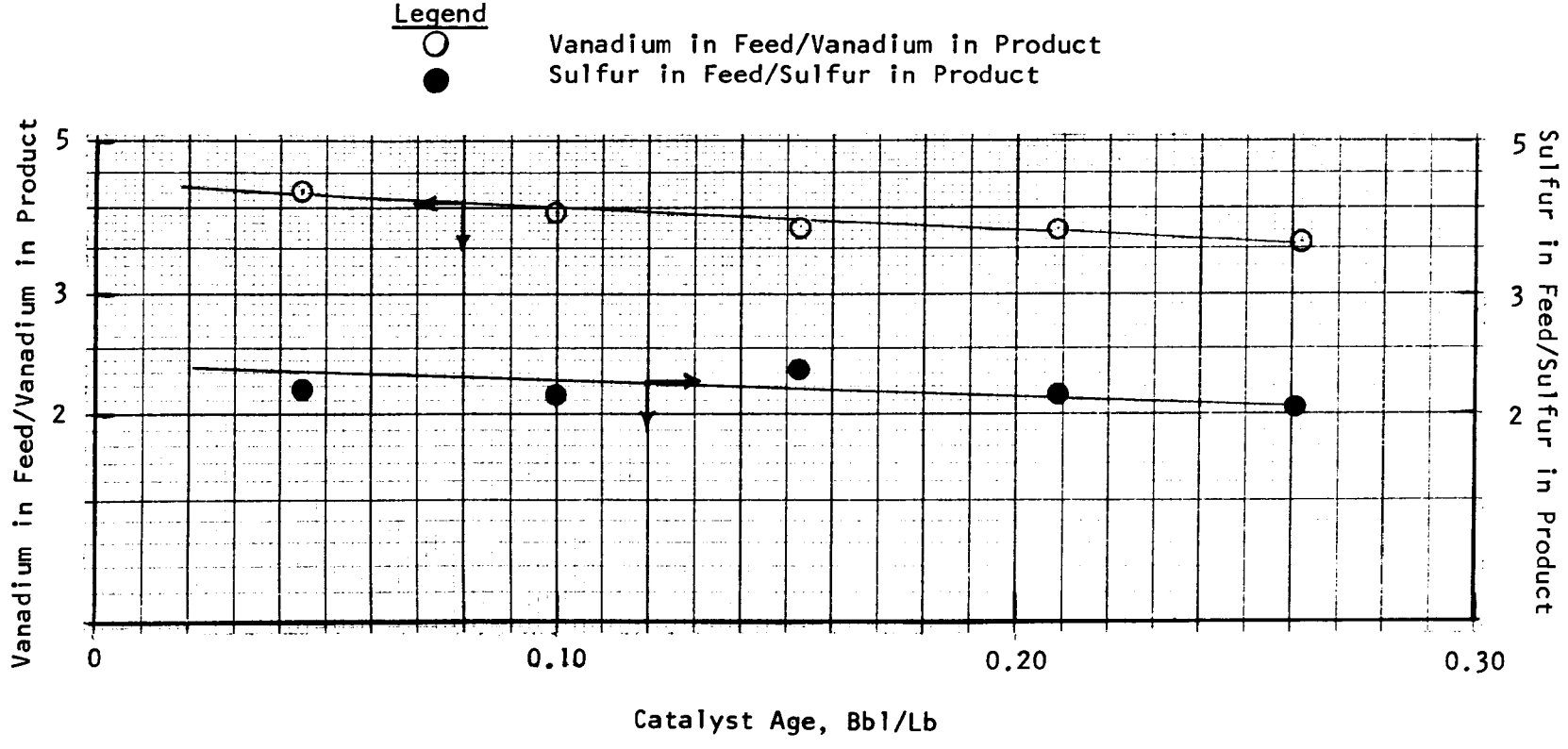
Figure 15. DEMETALLIZATION AND DESULFURIZATION OF BACHAQUERO VACUUM RESIDUUM

OVER 1.0 W % MOLYBDENUM/20 X 50 MESH BAUXITE

Run 185-236, Catalyst HRI 3634

Feed Composition	
Gravity, °API	5.3
Sulfur, W %	3.49
Vanadium, ppm	754
Nickel, ppm	96

Operating Conditions	
Hydrogen Pressure, psig	2000
Temperature, °F	790
Liquid Space Velocity, $V_O/Hr/V_r$	0.75
Catalyst Space Velocity, B/D/Lb	0.057
Hydrogen Rate, SCF/B (Vent)	4400



SECTION VI

DESULFURIZATION RUNS

DEMETALLIZATION FEEDSTOCK PREPARATION

Demetallized residua from each long term run on Tia Juana and Gach Saran were collected and blended so that a feed having constant properties could be fed to the desulfurization unit. The products that were blended to make the demetallized feed are listed in Table 5. Detailed inspections on the blended feeds for each of the desulfurization runs are given in Table 6.

CATALYST

The catalyst used for the desulfurization of the demetallized feeds was high activity American Cyanamid 0.02-inch beads. This same catalyst was used in the desulfurization step in Phase I of this contract. The properties of this catalyst are summarized in Table 7.

DESULFURIZATION OPERATING CONDITIONS

Desulfurization operating conditions were selected so that a maximum desulfurization with a minimum hydrogen consumption would be obtained. All runs were carried out in the fixed bed downflow reactor as previously described, except that the effective volume was reduced to half that used in the demetallization step. Because of the limited quantity of the demetallized feed available, the reduced volume would allow a better determination of catalyst aging characteristics. Each run was conducted at a hydrogen pressure of 2000 psig, a reactor temperature of 760°F, and a liquid space velocity of 1.0 $V_o/Hr/V_r$. Hydrogen rate was maintained at about 4500 SCF/B of feed. The

Table 5. COMPOSITION OF DEMETALLIZED RESIDUA

FED TO THE DESULFURIZATION REACTOR

Feed	Demetallized Tia Juana Vacuum Residuum	Demetallized Gach Saran Vacuum Residuum
HRI Identification Number	L-385	L-390
Products Blended to Make Composite Feed	Run 184-190 Period 2 to 24	Run 185-235 Period 2 to 28

Table 6. INSPECTIONS ON DEMETALLIZED VACUUM RESIDUUM FEEDSTOCKS

Demetallized Feedstock Source	Tia Juana Vacuum Residuum	Gach Saran Vacuum Residuum
HRI Identification No.	L-385	L-390
Demetallized Over	Commercial Demetallization Catalyst, HRI 3634	
Gravity, °API	12.8	13.1
Sulfur, W %	1.81	1.63
Ramsbottom Carbon, W %	13.6	12.1
Carbon, W %	87.07	87.16
Hydrogen, W %	10.92	11.00
H/C Atomic Ratio	1.49	1.50
Nitrogen, ppm	4900	5700
Vanadium, ppm	219	49
Nickel, ppm	53	38
Viscosity, SFS @ 210°F	72	57
IBP-650°F, V %	6.7	9.0
Gravity, °API	34.3	35.7
Sulfur, W %	0.35	0.35
650-975°F, V %	22.0	20.0
Gravity, °API	19.7	18.7
Sulfur, W %	1.14	0.79
975°F+, V %	71.3	71.0
Gravity, °API	8.6	8.6
Sulfur, W %	2.12	1.93
Ramsbottom Carbon, W %	19.0	16.6
Vanadium, ppm	295	59
Nickel, ppm	67	51

Table 7. SUMMARY OF INSPECTIONS ON AMERICAN CYANAMID 0.02" HIGH ACTIVITY BEADED CATALYST

HRI Identification Number	3104
Physical Properties	
Surface Area, M^2/g	250
H_2O Pore Volume, cc/g	0.67
Hg Pore Volume, cc/g	0.62
Screen Analysis, U.S. Sieve No.	
+20	1.3
20/30	16.9
30/40	76.2
40/50	5.0
50/70	0.5
70/100	0.1
-100	----
Chemical Analysis, W %	
MoO_3	(15.0)
CoO	(3.0)

runs were generally carried out to a catalyst age of 2.0 to 2.5 Bbl/Lb. Runs of this duration, together with results obtained during Phase I of this contract, provide an accurate measure of the catalyst deactivation rate which can be translated to the catalyst utilization required to obtain a given product desulfurization level. Detailed operating conditions and liquid product inspections for each run of this series are given in Appendix C.

DESULFURIZATION OF DEMETALLIZED FEEDS

The demetallized Tia Juana vacuum residuum was desulfurized in Runs 184-191 and 184-192. Run 184-191 was carried out to a catalyst age of 0.71 Bbl/Lb and was terminated because of an unusually high deactivation slope. Analyses of the spent catalyst after shutdown indicated no unusual loading of carbon, vanadium, or nickel. It was concluded that this abnormal behavior was due to an abnormal scatter in the analytical data.

Run 184-192 was a repeat of Run 184-191 and was carried out to a catalyst age of 1.9 Bbl/Lb. Desulfurization results for this run are summarized in Figure 16. Demetallized Gach Saran vacuum residuum was desulfurized in Run 184-193 to a catalyst age of 2.3 Bbl/Lb and the desulfurization results from this run are given in Figure 17.

Liquid product sulfurs obtained in this series of runs ranged from 0.49 to 0.65 weight percent for the Gach Saran feed and 0.6 to 0.85 weight percent for the Tia Juana feed. The operating conditions for these runs were such that correlations could be used to predict the operating conditions required for 0.5 weight percent fuel oil in an equilibrium catalyst situation. The results from these operations confirmed the low catalyst deactivation rates obtained in previous runs made under Phase I of the present project.

Figure 16. DESULFURIZATION OF DEMETALLIZED TIA JUANA VACUUM RESIDUUM

Run 184-192

Feed Composition	
Gravity, °API	12.8
Sulfur, W %	1.91
Vanadium, ppm	219
Nickel, ppm	53

Operating Conditions	
Hydrogen Pressure, psig	2000
Temperature, °F	760
Liquid Space Velocity, $V_F/Hr/V_R$	1.0
Catalyst Space Velocity, B/D/Lb	0.104
Hydrogen Rate, SCF/B (Vent)	4400

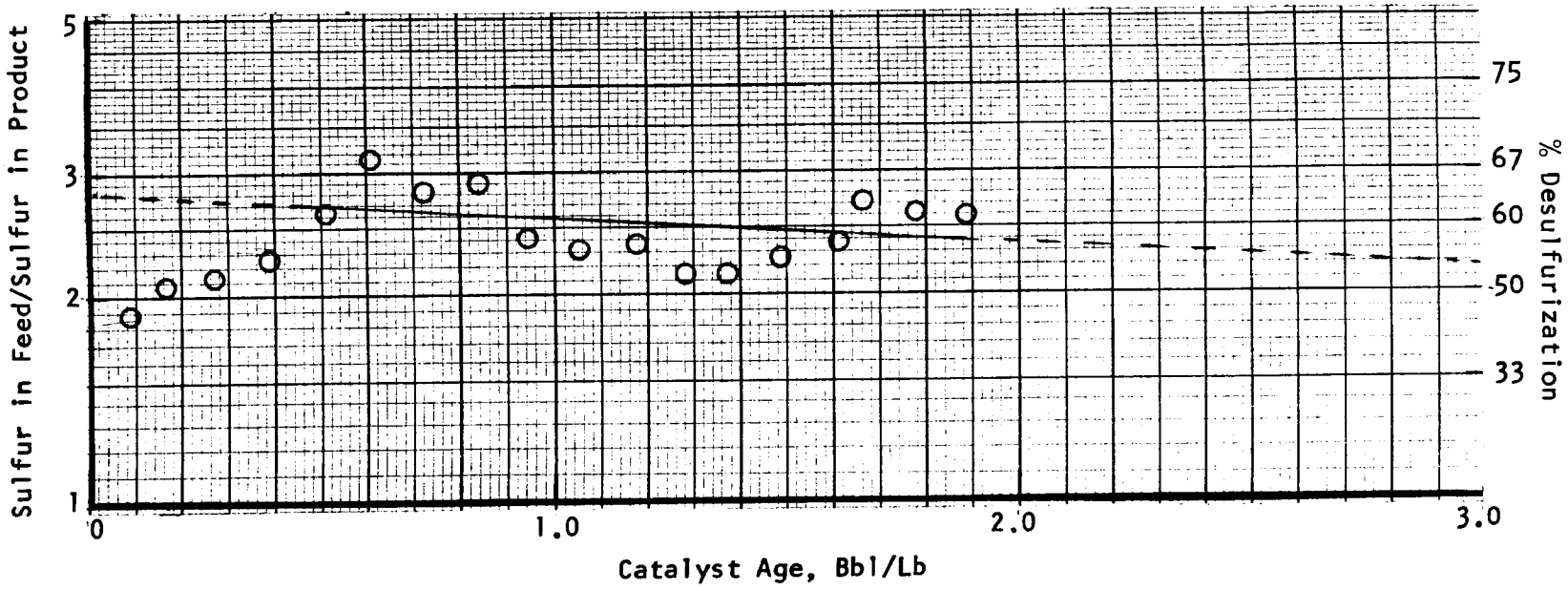
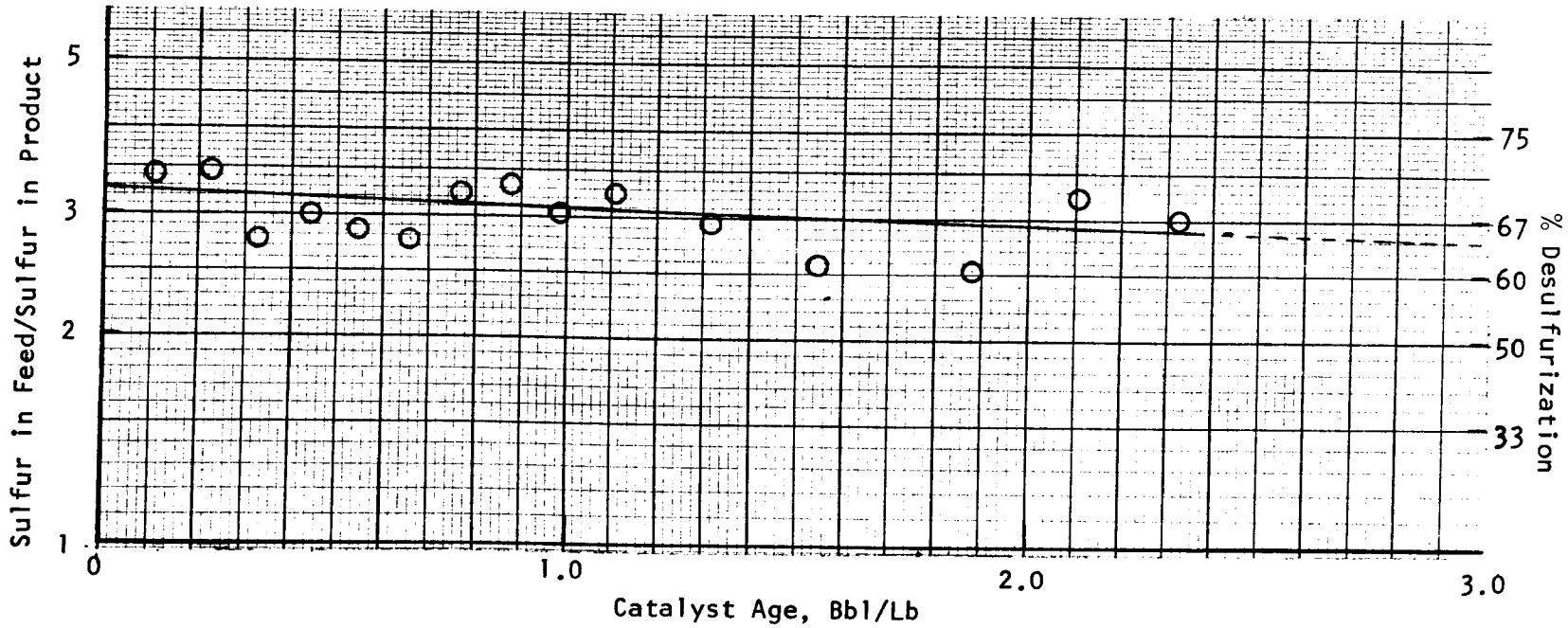


Figure 17. DESULFURIZATION OF DEMETALLIZED GACH SARAN VACUUM RESIDUUM

Run 184-193

Feed Composition	
Gravity, °API	13.1
Sulfur, W %	1.63
Vanadium, ppm	49
Nickel, ppm	38

Operating Conditions	
Hydrogen Pressure, psig	2000
Temperature, °F	760
Liquid Space Velocity, $V_F/Hr/V_R$	1.0
Catalyst Space Velocity, B/D/Lb	0.105
Hydrogen Rate, SCF/B (Vent)	4250



SPENT DESULFURIZATION CATALYST

A summary of analyses on samples of spent desulfurization catalysts is given in Table 8. The amounts of vanadium and nickel on the catalyst are much lower in comparison to those which would have been observed if the feeds had not been demetallized. It should also be noted that, at about the same catalyst age, the vanadium loading on the catalyst in the case of the Tia Juana feed is only about double that of the Gach Saran feed even though the vanadium content of the demetallized Tia Juana is about four times that of the Gach Saran feed.

The total level of metals in the demetallized feed gives no indication of the rate of deactivating the desulfurization catalyst. The determining factor is the ease with which the remaining metals can be removed from the demetallized products.

Metal balances for the two desulfurization runs are summarized in Table 9.

PRODUCT YIELDS AND INSPECTIONS

Detailed product inspections were obtained on one product from each of the desulfurization runs. The product taken for each run was about the middle of the run. Summaries of yields and product inspections are given in Tables D-3 and D-4 of Appendix D. An overall summary of the demetallization and desulfurization process to produce 0.5 weight percent 400°F+ fuel oil from Tia Juana and Gach Saran vacuum residua is given in Table 10. The 400°F+ fuel oil yield was about 97.6 volume percent for Tia Juana and 98.1 volume percent for Gach Saran. Naphtha yield was 7.5 volume percent for Tia Juana and 6.9 volume percent for Gach Saran.

Chemical hydrogen consumption in the desulfurization step ranged from about 310 SCF/B for demetallized Tia Juana to about 415 SCF/B for demetallized Gach Saran.

Table 8. ANALYSES OF SPENT DESULFURIZATION CATALYST

Run No.	Demetallized Vacuum Residuum Feed	Catalyst Age, Bbl/Lb	Weight Percent Element on Spent Catalyst			
			C	S	V	Ni
184-191	Tia Juana	0.7	14.25	4.38	0.99	0.28
184-192	Tia Juana	1.9	17.62	5.16	1.90	0.51
184-193	Gach Saran	2.3	17.52	5.20	1.05	0.76

Table 9. VANADIUM AND NICKEL BALANCES FROM DESULFURIZATION RUNS

Run Number	184-192		184-193	
Desulfurization Catalyst Age, Bbl/Lb	1.9		2.3	
Feed	Demetallized Tia Juana Vacuum Residuum		Demetallized Gach Saran Vacuum Residuum	
Feed Vanadium, ppm	219		49	
Feed Nickel, ppm	53		38	
	Grams	W % on Feed	Grams	W % on Feed
IN WITH THE FEED				
Vanadium	9.39		2.56	
Nickel	2.27		1.98	
VANADIUM OUT				
With Liquid Product	8.18	87.1	1.37	53.5
On Catalyst	1.84	19.5	0.98	38.3
Total	10.02	106.6	2.35	91.8
NICKEL OUT				
With Liquid Product	1.69	74.4	1.17	59.1
On Catalyst	0.49	21.6	0.71	35.8
Total	2.18	96.0	1.88	94.9

Table 10. SUMMARY OF RESULTS ON THE DEMETALLIZATION AND DESULFURIZATION OF VACUUM RESIDUA

(Feed and Product Analyses)

Vacuum Bottoms	Raw Feed	Demetallized Feed	Desulfurized Product
Tia Juana			
% Vanadium Removal		62	18
Sulfur, W %	2.9	1.81	0.5[1]
Vanadium, ppm	570	219	197[1]
Nickel, ppm	75	53	39[1]
C_4-400°F, V %	----	----	7.5[2]
400°F+, V %	----	----	97.6[2]
Gach Saran			
% Vanadium Removal		83	49
Sulfur, W %	3.72	1.63	0.5[1]
Vanadium, ppm	291	49	27[1]
Nickel, ppm	110	38	21[1]
C_4-400°F, V %	----	----	6.9[2]
400°F+, V %	----	----	98.1[2]

1. On 400°F+ Fraction
2. Volume % of Raw Feed

SECTION VII

PROCESS ECONOMICS

The major cost of producing low sulfur fuel oil from high metals residuum feeds depends on the cost of the facility necessary to carry out the demetallization and desulfurization operations, the amount of hydrogen consumed during the process, and the cost of the demetallization and desulfurization catalysts. Summaries of investment requirements and operating costs for producing 1.0, 0.5, and 0.3 weight percent sulfur fuel oil from Gach Saran and Tia Juana vacuum residua utilizing unpromoted bauxite and the commercially prepared 1.0 weight percent molybdenum on 20 x 50 mesh activated bauxite in the demetallization step and the commercial HDS beads in the desulfurization step are given in Tables 11 and 12, respectively.

The data computation for the 0.5 weight percent sulfur fuel oil product case requires very little extrapolation from the operating conditions utilized in the experimental program. For the 1.0 weight percent and 0.3 weight percent sulfur fuel oil cases, extrapolation of the data is necessary.

Results in Tables 11 and 12 indicated that the use of commercial 1.0 weight percent molybdenum catalyst in the demetallization step in place of activated bauxite contributed to a saving in investment costs of between $1.37 MM to $2.59 MM for the Tia Juana vacuum residuum feed and between $1.20 MM to $2.30 MM for the Gach Saran vacuum residuum feed (20,000 BPSD plant capacity). The saving in operating costs ranged from $0.05 per barrel to $0.07 per barrel for the Tia Juana feed and $0.04 per barrel to $0.07 per barrel for the Gach Saran feed.

For both feeds, the operating cost, as well as the investment costs, increased sharply as the level of sulfur in the fuel oil was reduced from 0.5 to 0.3 weight percent. The curves showing the variation of the operating cost for the Gach Saran and Tia Juana vacuum residua with the fuel oil sulfur level are given in Figure 18. The operating cost given in Tables 11 and 12 and

Table 11. INVESTMENT AND OPERATING COST FOR A TWO STAGE DEMETALLIZATION-DESULFURIZATION OPERATION OF TIA JUANA VACUUM RESIDUUM

BASES:
1. Plant Capacity - 20,000 BPSD
2. 1974 Gulf Coast Construction
3. Hydrogen Cost - $0.50/1000 SCF
4. Capital Charges - 25% of Investment Included in the Operating Cost
5. Activated Bauxite Cost - $0.10/Lb
6. Commercial 1% Mo Catalyst Cost - $0.23/Lb

Catalyst	Unpromoted Bauxite	Commercial 1% Mo Catalyst
1 W % Sulfur Fuel Oil		
Investment, MM$	15.92	13.33
Operating Cost, $/B	1.29	1.22
0.5 W % Sulfur Fuel Oil		
Investment, MM$	19.44	17.07
Operating Cost, $/B	1.69	1.63
0.3 W % Sulfur Fuel Oil		
Investment, MM$	20.15	18.78
Operating Cost, $/B	2.08	2.03

Table 12. INVESTMENT AND OPERATING COST FOR A TWO STAGE DEMETALLIZATION-DESULFURIZATION OPERATION OF GACH SARAN VACUUM RESIDUUM

BASES:
1. Plant Capacity - 20,000 BPSD
2. 1974 Gulf Coast Construction
3. Hydrogen Cost - $0.50/1000 SCF
4. Capital Charges - 25% of Investment Included in The Operating Cost
5. Activated Bauxite Cost - $0.10/Lb
6. Commercial 1% Mo Catalyst Cost - $0.23/Lb

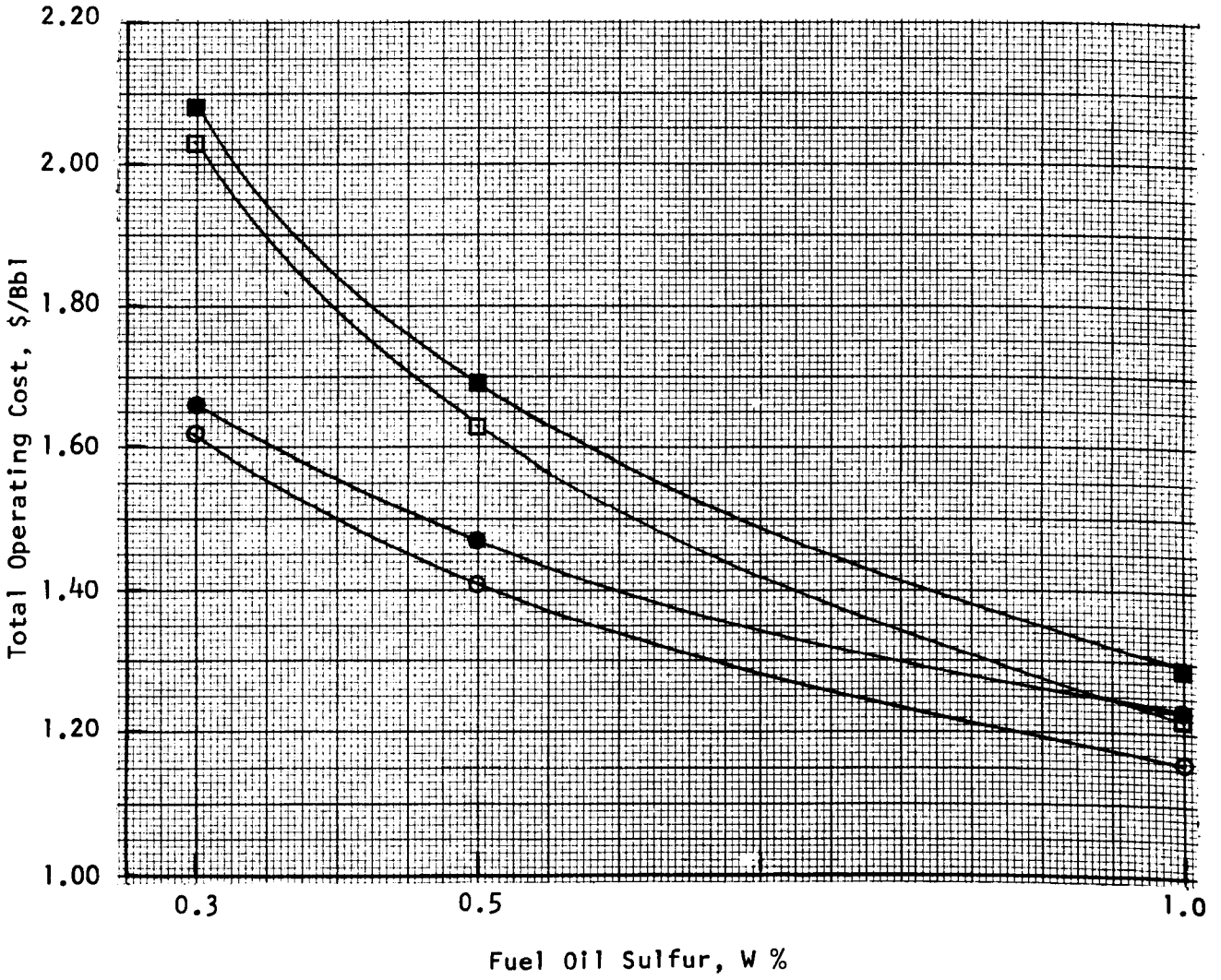
Catalyst	Unpromoted Bauxite	Commercial 1% Mo Catalyst
1 W % Sulfur Fuel Oil		
Investment, MM$	14.57	12.27
Operatinb Cost, $/B	1.23	1.16
0.5 W % Sulfur Fuel Oil		
Investment, MM$	17.72	16.06
Operating Cost, $/B	1.47	1.41
0.3 W % Sulfur Fuel Oil		
Investment, MM$	19.21	17.92
Operating Cost, $/B	1.66	1.62

Figure 18. TOTAL OPERATING COST

TWO STAGE DEMETALLIZATION-DESULFURIZATION

OF GACH SARAN AND TIA JUANA VACUUM RESIDUA

Symbol	Feed	Demetallization/Desulfurization Catalyst	Demetallization/Desulfurization Catalyst Cost, $/Lb
○	Gach Saran	Commercial 1% Mo/Beads	0.23/1.50
●	Gach Saran	Unpromoted Bauxite/Beads	0.10/1.50
□	Tia Juana	Commercial 1% Mo/Beads	0.23/1.50
■	Tia Juana	Unpromoted Bauxite/Beads	0.10/1.50



in Figure 18 include capital charges which are 25 percent of investment.

Estimated overall yield structure and product properties for the production of 400°F+ fuel oil containing 1.0, 0.5, and 0.3 weight percent sulfur from Gach Saran and Tia Juana vacuum residuum feeds are given in Tables 13 and 14, respectively.

Table 13. ESTIMATED OVERALL YIELDS AND PRODUCT PROPERTIES

CONSECUTIVE DEMETALLIZATION AND DESULFURIZATION OF GACH SARAN VACUUM RESIDUUM

400°F+ Fuel Oil Sulfur, W %	1.0				0.5				0.3			
Yields	W%	V%	°API	%S	W%	V%	°API	%S	W%	V%	°API	%S
H_2S & NH_3	3.2				3.9				4.1			
C_1-C_3	0.8				1.1				1.3			
C_4-400°F	3.6	5.0	63	<0.07	4.9	6.9	63	<0.07	6.1	8.5	63	<0.07
400-650°F	7.3	8.8	35	0.10	9.7	11.7	35	<0.07	11.8	14.2	35	<0.07
650-975°F	21.0	23.2	21	0.26	22.9	25.3	21.5	0.09	24.3	26.9	21.5	<0.07
975°F+	65.2	67.2	11.2	1.33	58.8	61.1	12.3	0.73	53.9	56.3	13	0.47
400°F+	93.5	99.2	15.3	1.0	91.4	98.1	17	0.5	90.0	97.4	18.2	0.3
TOTAL	101.1	104.2	17	0.96	101.3	105.0	19.4	0.47	101.5	105.9	21	0.28

Table 14. ESTIMATED OVERALL YIELDS AND PRODUCT PROPERTIES

CONSECUTIVE DEMETALLIZATION AND DESULFURIZATION OF TIA JUANA VACUUM RESIDUUM

400°F+ Fuel Oil Sulfur, W %	1.0				0.5				0.3			
Yields												
	W%	V%	°API	%S	W%	V%	°API	%S	W%	V%	°API	%S
H_2S & NH_3	2.3				2.9				3.2			
C_1-C_3	1.0				1.3				1.6			
C_4-400°F	4.2	5.8	62	<0.07	5.4	7.5	62	<0.07	6.5	9.0	62	<0.07
400-650°F	9.1	10.8	34	0.1	10.7	12.7	34	<0.07	12.7	15.1	34	<0.07
650-975°F	24.9	27.1	20	0.30	26.1	28.6	21.3	0.1	27.3	29.9	21.3	<0.07
975°F+	59.5	60.4	10	1.43	54.8	56.3	11.5	0.78	50.1	51.8	12.5	0.50
400°F+	93.5	98.3	15	1.0	91.6	97.6	16.9	0.5	90.1	96.8	18.2	0.3
TOTAL	101.0	104.1	17	0.96	101.2	105.1	19.4	0.47	101.4	105.8	21.1	0.28

SECTION VIII

APPENDICES

APPENDIX A

SUMMARY OF CATALYST SCREENING RUNS

Table A-1. SUMMARY OF CATALYST SCREENING RUNS

(Feed: Tia Juana Vacuum Bottoms)

Run No.	Period No.	Catalyst HRI No.	Catalyst Base	Catalyst Promoter	Preparation	Temp. °F	Hydrogen Pressure psig	Space Velocity $V_O/Hr/V_r$	Space Velocity B/D/Lb	Hydrogen Rate SCF/Bbl	Catalyst Age, Bbl/Lb	Product Inspections: Gravity °API	% S	ppm, V	ppm, Ni	IBP-550°F V /
184-182	1B	LX-28	Porocel	0.5% Mo	HRI Lab	789	2000	0.49	0.039	5203	0.038	14.7	1.94	145	42	5
	2		20 x 50 Mesh			791	2000	0.54	0.043	4328	0.070	14.2	1.69	141	47	7
	3					789	1990	0.55	0.044	4181	0.114	16.5	1.69	145	50	8
	4					790	2000	0.53	0.042	4230	0.156	15.2	1.54	138	49	9
	5					790	2005	0.50	0.040	3935	0.196	13.6	1.52	128	46	11
	6					790	2000	0.55	0.044	3566	0.240	14.6	1.49	141	50	19
	7					790	2010	0.53	0.042	3982	0.282	14.3	1.47	162	51	14
	8					789	1995	0.50	0.039	3784	0.321	14.6	1.48	147	49	10
	9					789	2040	0.53	0.042	3570	0.363	15.0	1.67	160	50	10
	10					791	2010	0.60	0.048	3732	0.411	14.9	1.63	155	47	10
	11					790	2025	0.53	0.042	4350	0.453	14.4	1.62	168	56	11
	12					790	2000	0.51	0.040	4178	0.504	15.1	1.59	162	55	10
	13					790	1990	0.51	0.041	4061	0.545	14.6	1.51	163	56	14
	14					788	1980	0.49	0.039	4240	0.584	14.0	1.75	191	49	10
	15					792	1985	0.52	0.041	4113	0.625	17.4	1.66	181	53	11
	16					791	2020	0.53	0.042	4116	0.667	15.9	1.73	181	57	7
	17					790	2030	0.51	0.041	4231	0.708	15.3	1.69	179	55	9
	18					791	2015	0.55	0.043	4118	0.751	14.1	1.39	199	57	11
	19					788	2000	0.52	0.041	4327	0.792	15.8	1.60	195	53	12
	20					790	2015	0.49	0.039	4143	0.831	17.8	1.61	194	45	11
	21					790	2000	0.51	0.041	4076	0.872	15.5	1.51	202	53	7
	22					791	2000	0.73	0.058	4049	0.330	12.9	1.70	257	58	8
	23					790	2000	0.75	0.059	4043	0.989	16.8	1.74	241	58	19
	24					790	2000	0.75	0.060	4048	1.049	14.2	1.93	235	56	5
	25					791	2000	0.52	0.041	4360	1.090	14.3	1.62	195	50	10
	26					790	2005	0.53	0.042	3551	1.132	14.4	1.64	203	52	13
	27					790	1990	0.51	0.040	3726	1.172	14.4	1.56	214	50	10
	28					790	2000	0.51	0.041	4246	1.213	14.7	1.68	214	53	9
	29					791	2000	0.51	0.041	5728	1.254	14.2	1.55	237	56	8
184-183	1B	3599	Porocel	2% Mo	Eng. Lab.	789	2000	0.55	0.041	4917	0.039	15.4	1.47	141	37	7
	2		20 x 50 Mesh			790	2000	0.49	0.037	5059	0.076	15.4	1.17	119	36	11
	3					789	2005	0.53	0.040	4320	0.129	15.7	1.09	134	40	8
	4					790	2010	0.51	0.038	4038	0.167	15.6	1.10	129	42	10
	5					790	2000	0.53	0.040	4095	0.204	15.6	1.11	140	48	10

NOTE: Eng. Lab. - Prepared by Engelhard in laboratory equipment.
Eng. Comm. - Prepared by Engelhard in commercial equipment.
HRI Lab - Prepared by Hydrocarbon Research in laboratory equipment.

Table A-1. SUMMARY OF CATALYST SCREENING RUNS

(Feed: Tia Juana Vacuum Bottoms)

Run No.	Period No.	Catalyst HRI No.	Catalyst Base	Catalyst Promoter	Preparation	Temp. °F	Hydrogen Pressure psig	Space Velocity $V_O/Hr/V_r$	Space Velocity B/D/Lb	Hydrogen Rate SCF/Bbl	Catalyst Age, Bbl/Lb	Product Inspections: Gravity °API	% S	ppm, V	ppm, Ni	IBP-550°F V %
184-184	1B	3596	Porocel	2/ Mo	Eng. Lab.	792	2000	0.60	0.045	3741	0.038	15.8	1.28	127	39	9
	2		20 x 50 Mesh			790	2005	0.51	0.039	4354	0.074	16.0	0.84	120	39	8
	3					789	2000	0.52	0.040	4118	0.114	15.9	1.14	133	44	8
	4					790	2000	0.52	0.040	3868	0.154	16.3	1.03	130	44	9
184-185	1B	3608	Porocel	1/ Mo	Eng. Lab.	790	2010	0.49	0.038	5050	0.037	14.0	1.45	129	38	8
	2		20 x 50 Mesh			790	2000	0.51	0.040	4122	0.077	16.8	1.31	123	39	9
	3					790	2000	0.47	0.037	4369	0.114	15.5	1.14	100	39	10
	4					789	2010	0.47	0.037	4227	0.151	16.2	1.05	99	44	9
184-186	1B	3610	Porocel	2/ Mo	Eng. Lab.	790	2000	0.76	0.058	4822	0.047	14.9	1.59	191	49	10
	2		20 x 50 Mesh			790	2000	0.75	0.057	4261	0.104	14.6	1.34	177	50	8
	3					789	2010	0.74	0.056	4012	0.160	14.4	1.17	178	50	7
	4					790	2000	0.75	0.057	3864	0.217	14.9	1.32	182	50	6
	5					789	2000	0.74	0.056	3834	0.273	14.8	1.21	184	50	8
	6					791	2000	0.75	0.057	4304	0.330	15.0	1.16	182	50	7
	7					787	1985	0.76	0.058	3640	0.388	15.5	1.21	221	53	7
	8					786	1975	0.55	0.042	3656	0.430	13.7	1.25	193	52	10
	9					789	1995	0.51	0.039	4107	0.469	15.2	1.06	151	47	12
	10					789	1985	0.50	0.038	4124	0.507	16.6	1.23	165	47	7
	11					790	2020	0.49	0.037	3935	0.544	15.3	1.27	179	48	8
	12					790	1995	0.52	0.039	3761	0.583	14.8	1.29	182	47	7
	13					790	2000	0.51	0.039	3906	0.622	15.4	1.28	176	49	7
	14					790	1995	0.51	0.039	3820	0.661	16.4	1.33	203	55	8
	15					790	2000	0.50	0.038	4061	0.699	15.3	1.30	198	49	8
	16					790	2005	0.49	0.037	4079	0.736	15.1	1.25	168	46	9
	17					791	2000	0.51	0.039	3851	0.775	14.8	1.23	191	49	9
	18					791	2015	0.51	0.039	4350	0.814	17.2	1.27	180	48	5
	19					790	2015	0.74	0.056	3877	0.870	14.0	1.54	238	51	7
	20					790	1985	0.75	0.057	3911	0.927	13.8	1.49	230	55	9
	21					789	1975	0.69	0.052	4355	0.979	14.8	1.40	227	55	9
	22					792	2000	0.75	0.057	3964	1.036	14.4	1.46	234	54	9
184-187	1B	LX-22-6	Porocel	2/ Mo	HRI Lab.	789	1985	0.79	0.060	3680	0.043	12.7	1.42	163	46	6
	2		20 x 50 Mesh			792	1980	0.76	0.058	4301	0.096	14.7	1.39	164	50	7
	3					793	2000	0.60	0.046	5303	0.142	13.8	1.30	168	50	7
	4					789	2000	0.75	0.057	4431	0.199	14.3	1.34	177	53	6

NOTE: Eng. Lab. - Prepared by Engelhard in laboratory equipment.
Eng. Comm. - Prepared by Engelhard in commercial equipment.
HRI Lab. - Prepared by Hydrocarbon Research in laboratory equipment.

Table A-1. SUMMARY OF CATALYST SCREENING RUNS

(Feed: Tia Juana Vacuum Bottoms)

Run No.	Period No.	Catalyst HRI No.	Catalyst Base	Catalyst Promoter	Preparation	Temp. °F	Hydrogen Pressure psig	Space Velocity $V_O/Hr/V_r$	Space Velocity B/D/Lb	Hydrogen Rate, SCF/Bbl	Catalyst Age Bbl/Lb	Product Inspections: Gravity °API	% S	ppm V	ppm Ni	IBP-550°F V %
184-188	1B	3630	Porocel	1% Mo	Eng. Lab.	790	2005	0.62	0.047	3750	0.038	14.4	1.21	155	39	5
	2		20 x 50 Mesh			789	2000	0.38	0.029	5307	0.063	17.2	0.91	122	36	10
	3					789	2010	0.46	0.036	4446	0.099	16.0	0.93	149	39	10
	4					791	2005	0.47	0.036	4413	0.135	16.8	0.88	129	40	9
	5					788	2000	0.51	0.039	4256	0.174	16.1	1.19	162	43	8
184-189	1B	3634	Porocel	1% Mo	Eng. Comm.	789	1995	0.47	0.036	4202	0.032	16.6	1.10	126	33	7
	2		20 x 50 Mesh			789	2000	0.54	0.041	3890	0.071	16.8	1.08	138	39	9
	3					790	2000	0.48	0.036	4537	0.107	16.2	1.07	136	39	9
	4					790	2000	0.52	0.039	4308	0.146	16.3	1.00	165	42	8
	5					790	2000	0.64	0.049	3266	0.195	16.3	1.12	187	45	8
185-224	1B	3581	Porocel	2% Mo	Eng. Lab.	791	1985	0.55	0.042	4898	0.034	17.1	1.18	101	30	9
	2		20 x 50 Mesh			790	2005	0.49	0.037	4769	0.068	16.7	0.87	79	33	9
	3					790	2000	0.48	0.037	5481	0.105	16.9	1.13	91	37	10
	4					792	2010	0.50	0.038	4914	0.140	16.5	1.00	99	39	11
185-225	1B	3582	Porocel	2% Mo	Eng. Lab.	787	1910	0.54	0.043	2473	0.036	13.2	1.41	141	41	7
	2		20 x 50 Mesh			789	2010	0.53	0.043	2541	0.081	15.2	1.58	134	39	11
	3					792	2030	0.47	0.038	5156	0.117	13.7	1.69	289	52	11
185-226	1B	3583	Porocel	2% Mo	Eng. Lab.	788	2005	0.50	0.037	4134	0.033	15.0	1.25	147	40	7
	2		20 x 50 Mesh			789	2010	0.51	0.038	6236	0.069	15.4	1.02	127	48	11
	3					789	1995	0.51	0.038	4989	0.107	15.1	1.06	138	49	10
	4					790	1970	0.45	0.034	4188	0.141	15.9	1.06	122	38	12
185-227	1B	3598	Porocel	2% Mo	Eng. Lab.	789	1995	0.51	0.039	6941	0.043	15.7	1.17	116	32	8
	2		20 x 50 Mesh			785	2020	0.54	0.042	4904	0.085	15.9	1.01	119	35	10
	3					788	2010	0.53	0.041	5085	0.126	16.1	1.03	123	38	9
	4					790	2010	0.53	0.041	4530	0.164	16.6	1.10	133	39	11
185-228	1B	3594	Porocel	2% Mo	Eng. Lab.	789	2000	0.60	0.045	3432	0.035	15.0	1.30	156	37	11
	2		20 x 50 Mesh			789	2000	0.53	0.041	4125	0.076	16.0	0.99	109	33	9
	3					790	2005	0.51	0.039	4443	0.103	16.4	0.90	117	37	9
	4					789	2000	0.53	0.041	4305	0.141	15.3	1.11	128	44	10
185-229	1B	3597	Porocel	2% Mo	Eng. Lab.	788	2010	0.55	0.043	3925	0.039	15.3	1.27	128	40	6
	2		20 x 50 Mesh			790	2000	0.52	0.040	5111	0.079	16.0	1.12	115	42	8
	3					790	2005	0.51	0.040	5784	0.119	15.9	1.01	116	39	8
	4					790	1990	0.50	0.039	3730	0.155	15.8	1.00	134	43	12

NOTE: Eng. Lab. - Prepared by Engelhard in laboratory equipment.
Eng. Comm. - Prepared by Engelhard in commercial equipment.
HRI Lab. - Prepared by Hydrocarbon Research in laboratory equipment.

Table A-1. SUMMARY OF CATALYST SCREENING RUNS

(Feed: Tia Juana Vacuum Bottoms)

Run No.	Period No.	Catalyst HRI No.	Catalyst Base	Catalyst Promoter	Preparation	Temp. °F	Hydrogen Pressure psig	Space Velocity V_O/Hr/V_r	Space Velocity B/D/Lb	Hydrogen Rate, SCF/Bbl	Catalyst Age, Bbl/Lb	Product Inspections: Gravity °API	Product Inspections: % S	Product Inspections: ppm, V	Product Inspections: ppm, Ni	Product Inspections: IBP-550°F V %
185-230	1B	3609	Porocel 20 x 50 Mesh	0.5% Mo	Eng. Lab.	791	2015	0.52	0.042	4259	0.042	14.7	1.59	159	41	8
	2					792	2010	0.52	0.042	4148	0.084	15.5	1.62	161	43	7
	3					790	2000	0.54	0.043	4048	0.127	13.4	1.28	141	45	9
	4					790	2025	0.46	0.037	4717	0.164	14.6	1.35	138	44	10
185-231	1B	3608	Porocel 20 x 50 Mesh	1% Mo	Eng. Lab.	786	1970	0.83	0.063	3644	0.049	15.4	1.68	189	48	5
	2					790	1990	0.75	0.057	3948	0.094	15.2	1.55	185	47	4
	3					788	2010	0.70	0.053	5475	0.147	14.6	1.51	194	49	4
	4					789	2000	0.79	0.060	4849	0.207	14.0	1.63	199	51	5
	5					792	1990	0.78	0.060	4873	0.267	14.3	1.49	198	50	5
	6					791	2000	0.78	0.059	3801	0.326	14.8	1.57	209	57	8
	7					790	2000	0.54	0.041	4800	0.367	14.9	1.40	163	44	6
	8					790	2000	0.49	0.038	4331	0.405	14.4	1.16	199	48	10
	9					790	2000	0.53	0.040	3906	0.445	15.4	1.23	156	46	8
	10					790	2005	0.53	0.040	5572	0.485	16.8	1.22	151	44	16
	11					790	2000	0.78	0.059	4687	0.544	13.7	1.43	203	49	7
	12					789	1990	0.74	0.056	4146	0.600	14.6	1.66	203	49	5
	13					788	1975	0.72	0.055	5342	0.655	14.7	1.48	202	51	6
	14					791	1995	0.84	0.064	3699	0.719	14.5	1.55	218	55	7
	15					791	2000	0.77	0.059	4473	0.778	14.2	1.59	201	52	6
	16					789	1995	0.76	0.058	4437	0.836	13.7	1.49	212	49	9
	17					790	1990	0.77	0.059	4231	0.895	14.1	1.58	222	52	6
	18					790	1995	0.75	0.057	4431	0.952	14.2	1.34	223	50	9
	19					789	2005	0.78	0.060	4336	1.012	14.2	1.53	225	52	6
	20					788	1990	0.77	0.059	3787	1.071	13.8	1.70	246	51	9
	21					791	2005	0.76	0.058	4383	1.129	13.5	1.75	232	60	9
	22					790	2000	0.79	0.060	4436	1.189	13.7	1.68	233	60	8
	23					791	1995	0.77	0.059	4107	1.248	12.9	1.73	228	56	9
	24					790	2000	0.78	0.059	4066	1.307	13.8	1.78	231	53	7
	25					790	2000	0.77	0.059	3899	1.366	14.0	1.85	231	53	7
	26					790	2000	0.78	0.059	4259	1.425	14.1	1.72	228	52	6
	27					790	1995	0.77	0.059	4076	1.479	14.5	1.17	230	54	8
185-233	1B	3635	Porocel 20 x 50 Mesh	1% Mo	Eng. Comm. Variation	788	2000	0.46	0.031	5626	0.039	17.6	1.25	97	29	6
	2					791	2000	0.56	0.038	3823	0.077	16.5	1.10	130	39	9
	3					791	2010	0.50	0.034	6569	0.111	16.0	1.09	131	39	8
	4					791	2010	0.51	0.034	5478	0.145	16.8	1.22	132	40	9
	5					791	2000	0.52	0.035	5994	0.180	15.9	1.20	143	39	7

NOTE: Eng. Lab. - Prepared by Engelhard in laboratory equipment.
Eng. Comm. - Prepared by Engelhard in commercial equipment.
HRI Lab. - Prepared by Hydrocarbon Research in laboratory equipment.

APPENDIX B

SUMMARY OF DEMETALLIZATION RUNS

Table B-1. SUMMARY OF DEMETALLIZATION RUNS

Run No.-Period	Catalyst HRI No.	Catalyst Base	Catalyst Promoter	Preparation	Feed	Temp. °F	H_2 Pres. psig	Space Velocity V_o/Hr/Vr	Space Velocity B/D/Lb	H_2 Rate SCF/Bbl	Cat. Age, Bbl/Lb	Product Inspections: Gravity °API	% S	V ppm	Ni ppm	IBP-550°F V %
184-190-1B	3634	Porocel	1% Mo	Engelhard	Tia Juana	793	2010	0.90	0.067	3232	0.050	15.5	1.69	194	45	5
2		20 x 50 Mesh		Commercial	Vac. Btms.	788	2025	0.79	0.059	3833	0.109	13.8	1.49	198	46	6
3						790	2010	0.75	0.056	4155	0.165	14.6	1.48	191	45	5
4						790	1990	0.63	0.047	4670	0.212	14.9	1.43	185	47	7
5						790	1995	0.71	0.053	4737	0.265	14.9	1.42	195	50	7
6						792	2000	0.79	0.059	4306	0.324	14.9	1.53	201	50	7
7						792	2005	0.81	0.060	4031	0.384	14.2	1.58	226	56	6
8						790	2025	0.82	0.061	3949	0.445	13.6	1.67	226	56	7
9						794	2000	0.71	0.053	4626	0.498	14.3	1.63	224	50	6
10						791	2015	0.77	0.057	3932	0.555	14.1	1.53	219	48	6
11						792	2015	0.72	0.054	4081	0.609	14.9	1.58	210	49	7
12						791	2015	0.72	0.053	4438	0.662	15.8	1.45	210	49	7
13						790	2030	0.73	0.054	4530	0.716	15.1	1.60	222	49	7
14						792	2030	0.75	0.056	4328	0.772	14.2	1.57	235	50	5
15						791	2010	0.74	0.055	4204	0.827	14.4	1.57	221	49	7
16						790	2015	0.74	0.055	4252	0.882	14.1	1.68	252	49	4
17						791	2035	0.78	0.058	4076	0.940	14.1	1.69	248	51	5
18						792	2025	0.74	0.055	4253	0.995	13.0	1.58	242	50	7
19						792	2005	0.80	0.059	3830	1.054	13.4	1.44	262	52	7
20						790	2005	0.79	0.059	3853	1.113	13.2	1.64	259	50	6
21						790	2010	0.74	0.055	4055	1.168	14.0	1.56	268	52	5
22						789	2005	0.77	0.057	3888	1.225	13.8	1.80	278	57	5
23						790	2000	0.77	0.057	3922	1.282	13.8	1.79	265	54	4
24						789	2005	0.76	0.057	4104	1.339	13.4	1.99	272	54	6
185-235-1B	3634	Porocel	1% Mo	Engelhard	Gach Saran	788	1980	0.75	0.057	3551	0.050	15.7	1.56	32	27	5
2		20 x 50 Mesh		Commercial		790	2023	0.84	0.064	4399	0.114	15.3	1.53	35	29	7
3						786	2008	0.83	0.063	6099	0.177	15.1	1.58	37	33	8
4						787	2010	0.79	0.060	4726	0.237	14.9	1.54	36	39	6
5						789	1995	0.77	0.058	4504	0.295	14.6	1.45	36	36	6
6						788	1985	0.72	0.055	4816	0.350	15.3	1.57	37	38	6
7						790	1995	0.75	0.057	6125	0.407	15.0	1.50	33	37	6
8						787	1988	0.76	0.058	5099	0.465	14.6	1.53	39	40	6
9						786	1970	0.74	0.056	3894	0.521	14.5	1.79	36	38	6
10						790	1977	0.80	0.060	5683	0.581	13.7	1.76	40	41	5

Table B-1. SUMMARY OF DEMETALLIZATION RUNS

Run No.-Period	Catalyst HRI No.	Catalyst Base	Catalyst Promoter	Preparation	Feed	Temp. °F	H_2 Pres. psig	Space Velocity $V_o/Hr/V_r$	Space Velocity B/D/Lb	H_2 Rate SCF/Bbl	Cat. Age, Bbl/Lb	Product Inspections: Gravity °API	% S	V ppm	Ni ppm	IBP-550°F V %
185-235-11	3634	Porocel 20 x 50 Mesh	1/ Mo	Engelhard Commercial	Gach Saran	789	1985	0.72	0.054	4515	0.635	14.3	1.65	39	35	8
12						792	2010	0.76	0.058	4364	0.693	14.4	1.66	44	35	7
13						790	2000	0.81	0.061	5623	0.754	14.6	1.66	44	38	8
14						790	1975	0.75	0.057	3720	0.811	14.7	1.56	44	37	6
15						790	1984	0.79	0.059	3913	0.870	14.8	1.61	48	37	5
16						790	1998	0.75	0.057	4478	0.927	14.6	1.72	51	37	6
17						789	1989	0.71	0.054	6324	0.981	15.3	1.66	49	38	9
18						789	2007	0.74	0.056	3943	1.037	14.8	1.60	44	38	8
19						790	2003	0.77	0.058	4922	1.095	14.4	1.71	51	41	13
20						789	2000	0.72	0.054	5510	1.149	14.0	1.64	52	41	12
21						789	1993	0.73	0.055	4153	1.204	14.9	1.68	52	40	9
22						791	2000	0.76	0.058	4405	1.262	14.6	1.60	52	41	6
23						790	1983	0.60	0.045	3718	1.281	15.7	1.61	54	38	6
24						790	1990	1.00	0.076	2935	1.357	14.4	1.55	52	39	9
25						789	1988	0.71	0.054	6055	1.411	14.9	1.54	61	40	9
26						790	1993	0.76	0.058	4573	1.469	13.5	1.70	63	41	7
27						791	2018	0.86	0.065	3941	1.534	13.9	1.65	63	41	7
28						787	2008	0.76	0.058	5833	1.590	14.2	1.78	64	43	7
185-236-1B	3634	Porocel 20 x 50 Mesh	1/ Mo	Engelhard Commercial	Bachaquero	788	1983	0.68	0.051	4191	0.045	15.2	1.52	167	46	6
2						792	1986	0.72	0.053	4400	0.100	14.8	1.58	184	51	8
3						789	1983	0.74	0.055	4178	0.153	15.4	1.48	199	52	8
4						790	1981	0.75	0.056	4040	0.209	14.6	1.61	202	60	7
5						789	1998	0.72	0.053	5145	0.262	14.8	1.63	204	59	8

APPENDIX C

SUMMARY OF DESULFURIZATION RUNS

Table C-1. SUMMARY OF DESULFURIZATION RUNS

Run No.-Period	Catalyst HRI No.	Catalyst Base	Demetallized Feed	Demetallized Over	Temp. °F	Hydrogen Pressure psig	Space Velocity V_O/Hr/Vr	Space Velocity B/D/Lb	Hydrogen Rate SCF/Bbl	Catalyst Age Bbl/Lb	Product Inspections Gravity °API	% S	V ppm	Ni ppm	IBP-550°F V %
184-191-1B	3104	Amer. Cy.	Tia Juana	Comm. Demet.	757	2000	1.00	0.109	3339	0.083	16.7	0.67	176	39	7
2		0.02" Beads	Vac. Btms.	Catalyst	761	2018	1.11	0.119	3208	0.202	17.4	0.76	166	40	5
3				HRI 3634	758	2020	1.00	0.107	3901	0.309	16.4	0.97	169	41	4
4					759	2008	1.03	0.109	3929	0.418	17.0	0.83	193	40	6
5					761	1992	1.08	0.115	3958	0.533	17.2	0.91	191	39	6
6					763	1983	1.01	0.108	4026	0.641	16.8	0.90	189	39	6
7					761	1984	0.68	0.073	5620	0.714	16.7	0.82	191	40	5
184-192-1B	3104	Amer. Cy.	Tia Juana	Comm. Demet.	757	1998	1.01	0.105	4753	0.099	15.9	0.96	195	36	4
2		0.02" Beads	Vac. Btms.	Catalyst	758	1980	0.96	0.100	4983	0.174	16.9	0.86	193	36	5
3				HRI 3634	762	2007	0.99	0.103	4964	0.277	17.0	0.85	193	38	5
4					759	1988	1.08	0.112	4319	0.389	16.1	0.83	197	41	5
5					762	2005	1.12	0.116	4619	0.505	16.1	0.74	198	40	9
6					760	1997	1.08	0.112	4546	0.617	15.6	0.61	194	41	6
7					760	1968	1.00	0.104	4736	0.721	16.1	0.64	194	39	7
8					760	1993	1.17	0.122	3969	0.843	16.4	0.69	189	43	6
9					760	2012	1.02	0.106	4419	0.949	16.6	0.77	190	42	5
10					761	2007	1.04	0.108	4386	1.057	16.3	0.80	194	42	8
11					758	2002	1.22	0.127	3563	1.184	16.0	0.85	196	41	5
12					759	1989	0.95	0.098	4820	1.282	16.3	0.81	192	43	6
13					760	1987	0.91	0.094	5225	1.376	16.4	0.79	190	43	7
14					759	2002	1.17	0.122	3834	1.498	16.7	0.86	207	43	6
15					761	2015	1.11	0.116	3895	1.614	16.8	0.81	206	42	5
16					763	2010	1.14	0.119	3758	1.664	17.4	0.72	199	39	6
17					760	2029	1.13	0.117	4012	1.781	16.3	0.73	206	37	6
18					761	2032	1.08	0.112	4290	1.893	16.2	0.73	207	41	6
184-193-1B	3104	Amer. Cy.	Gach Saran	Comm. Demet.	758	1980	1.08	0.113	5339	0.117	17.2	0.50	30	20	4
2		0.02" Beads	Vac. Btms.	Catalyst	761	1977	1.06	0.111	4928	0.233	16.8	0.49	29	19	6
3				HRI 3634	761	2002	1.01	0.106	4010	0.335	16.6	0.59	30	19	5
4					759	1992	1.04	0.109	4253	0.444	16.8	0.55	29	22	5
5					759	2000	1.02	0.107	4220	0.551	17.2	0.58	29	23	5
6					759	2018	1.04	0.109	4061	0.660	17.5	0.60	30	24	5
7					761	2000	1.06	0.111	4628	0.771	18.2	0.52	28	23	5
8					760	2020	1.04	0.109	4652	0.880	16.4	0.50	28	23	5
9					758	2118	1.05	0.111	4422	0.991	16.8	0.56	28	23	5
10					758	2018	1.07	0.112	4142	1.103	16.4	0.52	29	22	5
11					763	2002	1.07	0.112	4098	1.215	*	*	*	*	*
12					762	2004	1.04	0.109	4287	1.324	17.7	0.56	24	24	7
13					762	1993	1.07	0.112	4067	1.436	*	*	*	*	*
14					760	2015	1.05	0.110	4027	1.546	17.9	0.65	25	25	5
15					760	2020	1.06	0.111	4095	1.657	*	*	*	*	*
16					760	2000	1.11	0.117	4511	1.774	*	*	*	*	*
17					760	2035	1.03	0.108	4663	1.882	17.4	0.66	26	25	6

Amer. Cy. = American Cyanamid

* No analyses were conducted on the products.

Table C-1. SUMMARY OF DESULFURIZATION RUNS

Run No.-Period	Catalyst HRI No.	Catalyst Base	Demetallized Feed	Demetallized Over	Temp. °F	Hydrogen Pressure psig	Space Velocity $V_o/Hr/V_r$	Space Velocity B/D/Lb	Hydrogen Rate SCF/Bbl	Catalyst Age Bbl/Lb	Product Inspections: Gravity °API	% S	V ppm	Ni ppm	IBP-550°F V %
184-193-18	3104	Amer. Cy.	Gach Saran	Comm. Demet.	760	2000	1.08	0.113	4066	1.995	*	*	*	*	*
19		0.02" Beads	Vac. Btms.	Catalyst	762	2010	1.07	0.112	4141	2.107	16.6	0.53	*	*	4
20				HRI 3634	759	2010	1.00	0.105	4134	2.212	*	*	*	*	*
21					760	2010	1.08	0.113	3945	2.325	16.0	0.57	*	*	3
201-83- 1B	3104	Amer. Cy.	Tia Juana	LX-28	763	2000	1.18	0.124	4661	0.097	16.7	0.59			7[a]
2		0.02" Beads	Vac. Btms.	(0.5% Mo)	760	2010	1.00	0.105	4961	0.174	17.0	0.52	115	40	7
3					760	2000	1.08	0.113	4269	0.287	16.6	0.45			6
4					760	2005	1.22	0.128	3792	0.415	16.0	0.53	116	38	6
5					760	1990	1.06	0.111	4425	0.526	16.0	0.59	121	38	7
6					761	1990	0.97	0.102	4687	0.628	15.8	0.60	118		7
7					757	1990	0.95	0.101	4888	0.729	16.1	0.57		38	6
8					760	2000	1.03	0.108	4122	0.829	16.0	0.57	112	37	7

Amer. Cy. = American Cyanamid

* No analyses were conducted on the products.

a. IBP-600°F for Run 201-83.

APPENDIX D

OPERATING CONDITIONS, YIELDS, AND PRODUCT PROPERTIES

Table D-1. OPERATING CONDITIONS, YIELDS, AND PRODUCT PROPERTIES

Run Number	184-190 Composite
Catalyst Age, Bbl/Lb	0.05-1.34
Feed	Tia Juana Vacuum Residuum
HRI Identification No.	3615
Catalyst	Comm. Demet. Catalyst (20 x 50 Mesh Porocel - 1% Mo)
HRI No.	3634

OPERATING CONDITIONS

Hydrogen Pressure, psig	2010
Temperature, °F	791
Liquid Space Velocity, V/Hr/V	0.76
Catalyst Space Velocity, B/D/Lb	0.057
Hydrogen Rate, SCF/B (Vent)	4100
Reactor Type	Downflow
Hydrogen Consumption, SCF/B	(350)
975°F+ Conversion, V %	17.6

YIELDS

	W%	V%
H_2S & NH_3	1.4	
C_1-C_3	0.6*	
C_4-C_6	0.4*	0.7*
IBP-500°F	2.0	2.5
500-650°F	3.9	4.5
650°F+	92.2	94.7
650-975°F	20.3	22.2
975°F+	71.9	72.5
Total	100.5	102.4
C_4+		
Gravity, °API		
Sulfur, W %		
Vanadium/Nickel		

	13.2
	1.80
	218/53

FRACTION, °F	Coll. Liq.	IBP-500	500-650	650+	650-975	975+
V % on Feed	101.4	2.5	4.5	94.7	22.2	72.5
Gravity, °API	12.8	30.3	29.9	11.1	19.8	8.6
Sulfur, W %	1.81	0.11	0.51	(1.87)	1.00	2.12
Carbon, W %	87.14					
Hydrogen, W %	10.96					
H/C Atomic Ratio	1.50					
Nitrogen, ppm	4900					
Bromine No., cgs/gm		10.6	13.5			
Aniline Point, °F			143		174	
Pour Point, °F				110	75	
Flash Point, °F				585		
ASTM Color						
RCR, W %						19.0
Vanadium ppm	219					295
Nickel, ppm	53					67
Viscosity, SUS @ 210°F					286	
, SFS @ 210°F				185		

* Calculated from correlations

Table D-2. OPERATING CONDITIONS, YIELDS, AND PRODUCT PROPERTIES

Run Number	185-235-14
Catalyst Age, Bbl/Lb	0.81
Feed	Gach Saran Vacuum Residuum
HRI Identification No.	3574
Catalyst	Comm. Demet. Catalyst (20 x 50 Mesh Porocel - 1% Mo)
HRI No.	3634

OPERATING CONDITIONS

Hydrogen Pressure, psig	1975
Temperature, °F	790
Liquid Space Velocity, V/Hr/V	0.75
Catalyst Space Velocity, B/D/Lb	0.057
Hydrogen Rate, SCF/B (Vent)	3700
Reactor Type	Downflow
Hydrogen Consumption, SCF/B	515
975°F+ Conversion, V %	20.9

YIELDS

	W%	V%
H_2S & NH_3	2.5	
C_1-C_3	0.8**	
C_4-C_6	0.4**	0.7**
IBP-500°F	3.4	4.3
500-650°F	3.7	4.6
650°F+	90.0	93.0
650-975°F	17.2	18.6
975°F+	72.8	74.4
Total	100.8	102.6
C_4+		
Gravity, °API	14.1	
Sulfur, W %	1.49	
Vanadium/Nickel	44/37	

FRACTION, °F	Coll. Liq.	IBP-500	500-650	650+	650-975	975+
V % on Feed	101.9	4.3	4.6	93.0	18.6	74.4
Gravity, °API	13.7	41.2	38.5	10.7	17.6	8.9
Sulfur, W %	1.50	0.02	0.37	1.61	0.84	1.78
Carbon, W %	86.80					
Hydrogen, W %	11.04					
H/C Atomic Ratio	1.07					
Nitrogen, ppm	6200					
Bromine No., cgs/gm		12.5	14.4			
Aniline Point, °F			136		163	
Pour Point, °F				105	90	
Flash Point, °F				550		
RCR, W %						17.3
Vanadium, ppm	44					59
Nickel, ppm	37					51
Viscosity, SUS @ 122°F					*	
, SFS @ 210°F				132		

* Insufficient sample
** Calculated from correlations

Table D-3. OPERATING CONDITIONS, YIELDS, AND PRODUCT PROPERTIES

Run Number	184-192-8
Catalyst Age, Bbl/Lb	0.84
Feed	Demetallized Tia Juana Vacuum Residuum
HRI Identification No.	L-385
Demetallized Over	Comm. Demet. Catalyst (1% Moly, HRI 3634)
Catalyst	American Cyanamid 0.02" Beads
HRI No.	3104

OPERATING CONDITIONS

Hydrogen Pressure, psig	1990
Temperature, °F	760
Liquid Space Velocity, V/Hr/V	1.17
Catalyst Space Velocity, B/D/Lb	0.122
Hydrogen Rate, SCF/B (Vent)	4000
Reactor Type	Downflow
Hydrogen Consumption, SCF/B	310
975°F+ Conversion, V %	10.0

YIELDS

	W%	V%
H_2S & NH_3	1.4	
C_1-C_3	0.7	
C_4-C_6	0.5*	0.8*
IBP-500°F	2.5	3.0
500-650°F	5.9	6.7
650°F+	89.5	90.1
650-975°F	24.5	25.9
975°F+	65.0	64.2
Total	100.5	100.6
C_4+		
Gravity, °API	16.0	
Sulfur, W %	0.69	
Vanadium/Nickel	188/43	

FRACTION, °F	Coll. Liq.	IBP-500	500-650	650+	650-975	975+
V % on Feed	99.8	3.0	6.7	90.1	25.9	64.2
Gravity, °API	15.5	39.8	31.1	13.5	21.1	11.1
Sulfur, W %	0.69	<0.02	<0.02	0.86	0.22	0.97
Carbon, W %	87.47					
Hydrogen, W %	11.28					
H/C Atomic Ratio	1.54					
Nitrogen, ppm	3720					
Bromine No., cgs/gm		2.1	5.9			
Aniline Point, °F			141		171	
Pour Point, °F				75	70	
Flash Point, °F				500		
ASTM Color					D8.0	
RCR, W %						16.7
Vanadium, ppm	189					315
Nickel, ppm	43					58
Viscosity, SUS @ 210°F					226	
, SFS @ 210°F				88		

* Calculated from correlations

Table D-4. OPERATING CONDITIONS, YIELDS, AND PRODUCT PROPERTIES

Run Number	184-193-15
Catalyst Age, Bbl/Lb	1.66
Feed	Demetallized Gach Saran Vacuum Residuum
HRI Identification No.	L-390
Demetallized Over	Comm. Demet. Catalyst (1% Moly, HRI 3634)
Catalyst	American Cyanamid 0.02" Beads
HRI No.	3104

OPERATING CONDITIONS

Hydrogen Pressure, psig	2020
Temperature, °F	760
Liquid Space Velocity, V/Hr/V	1.06
Catalyst Space Velocity, B/D/Lb	0.111
Hydrogen Rate, SCF/B (Vent)	4100
Reactor Type	Downflow
Hydrogen Consumption, SCF/B	415
975°F+ Conversion, V %	7.6

YIELDS

	W%	V%
H_2S & NH_3	1.4	
C_1-C_3	0.8	
C_4-C_6	0.5	0.9
IBP-500°F	2.8	3.3
500-650°F	5.6	6.3
650°F+	89.5	90.7
650-975°F	23.7	25.1
975°F+	65.8	65.6
Total	100.6	101.2
C_4+		
Gravity, °API	17.2	
Sulfur, W %	0.51	

FRACTION, °F	Coll. Liq.	IBP-500	500-650	650+	650-975	975+
V % on Feed	100.3	3.3	6.3	90.7	25.1	65.6
Gravity, °API	16.7	38.6	29.9	13.8	19.7	11.0
Sulfur, W %	0.51	<0.02	0.07	0.57	0.16	0.66
Carbon, W %	88.09					
Hydrogen, W %	11.54					
H/C Atomic Ratio	1.56					
Nitrogen, ppm	3900					
Bromine No., cgs/gm		3.5	5.5			
Pour Point, °F				80	90	
RCR, W %						13.5
Viscosity, SUS @ 122°F					204	
, SFS @ 210°F				54		

APPENDIX E

CONVERSION TABLE

APPENDIX G

CONVERSION TABLE

Variable	British Units	Metric Units	Conversion Factor
Temperature	Degrees Fahrenheit, °F	Degrees Centigrade, °C	$°C = 5/9(°F-32)$
Pressure	Pounds per Square Inch Gauge, psig	Kilograms per Square Centimeter, Kg/cm^2	$Kg/cm^2 = \frac{psig}{14.22}$
Hydrogen Rate	Standard Cubic Feet per Barrel, (60°F, 1 Atm.)	Normal Cubic Meters per Cubic Meter, NM^3/M^3 (0°C, 760 mm Hg)	$NM^3/M^3 = 0.168(SCF/Bbl)$

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

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EPA-650/2-73-041-a		

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	6. PERFORMING ORGANIZATION CODE

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M. C. Chervenak, P. Maruhnic, and G. Nongbri	

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15. SUPPLEMENTARY NOTES

16. ABSTRACT

The report gives Phase II results of a study of demetallization of heavy residual oils. Phase I was an experimental laboratory investigation to find a new low-cost demetallization catalyst for high metals, high sulfur residual oils. Phase II utilized the Phase I results to test the effectiveness of a demetallization catalyst when prepared on a commercial scale. The commercial production catalyst was tested for activity and aging characteristics and compared to laboratory prepared catalysts. The report includes descriptions of the catalyst, test units, and operating conditions and procedures.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group	
Air Pollution	Nickel	Air Pollution Control	13B	
Residual Oils	Contaminants	Stationary Sources	11H, 21D	
Hydrogenation	Catalysts	Demetallization	07C	07D
Metals	Scavengers (Materials)	Promoter	11F, 07B	11G
Vanadium	Fossil Fuels	Pretreatment		
Sulfur	Desulfurization	Clean Fuels		07A

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