

AD-A182 333

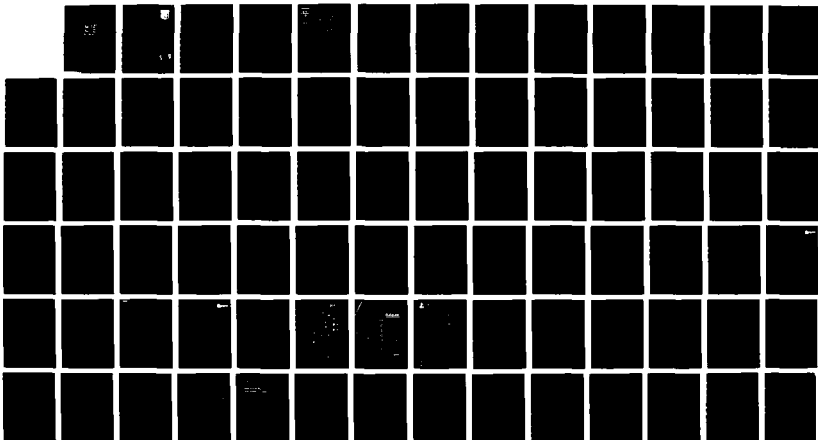
PRODUCTION OF HIGH ENERGY AVIATION FUELS FROM ADVANCED
COAL LIQUIDS PHASE 1(U) STRAT CO SALT LAKE CITY UT
J DOWNEN APR 87 AFMAL-TR-87-2036 F33615-86-C-2668

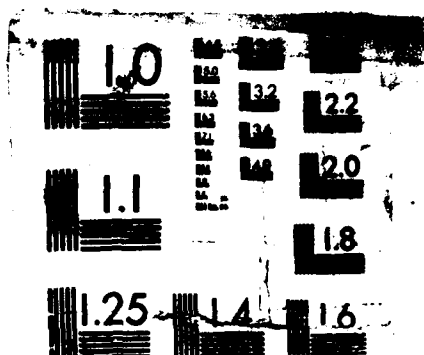
1/1

UNCLASSIFIED

F/G 21/2

NL





MICROCOPY RESOLUTION TEST CHART

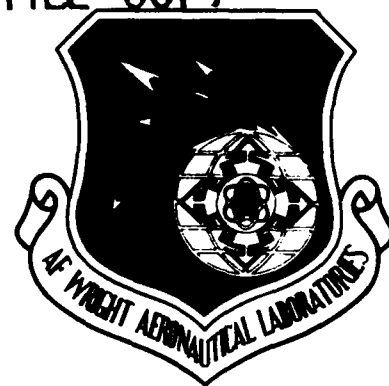
AD-A182 333

DTIC FILE COPY

2

AFWAL-TR-87-2036

PRODUCTION OF HIGH ENERGY AVIATION FUELS
FROM ADVANCED COAL LIQUIDS



JOHN DOWNEN

STRAT CO.
4597 JUPITER DRIVE
SALT LAKE CITY, UTAH 84124

APRIL 1987

FINAL REPORT FOR THE PERIOD JUNE 1986 - FEBRUARY 1987

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

DTIC
ELECTE
JUL 02 1987
S **D**
E

AERO PROPULSION LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433-6563

87

167

NOTICE

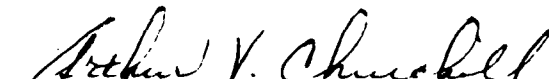
When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report has been reviewed by the Office of Public Affairs (ASD/PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.



WILLIAM E. HARRISON III
Fuels Branch
Fuels and Lubrication Division
Aero Propulsion Laboratory



ARTHUR V. CHURCHILL, Chief
Fuels Branch
Fuels and Lubrication Division
Aero Propulsion Laboratory

FOR THE COMMANDER



ROBERT D. SHERRILL, Chief
Fuels and Lubrication Division
Aero Propulsion Laboratory

"If your address has changed, if you wish to be removed from our mailing list, or if the addressee is not longer employed by your organization, please notify AFWAL/POSF, Wright-Patterson AFB, Ohio 45433-6563 to help us maintain a current mailing list."

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

DISCLAIMER NOTICE

**THIS DOCUMENT IS BEST QUALITY
PRACTICABLE. THE COPY FURNISHED
TO DTIC CONTAINED A SIGNIFICANT
NUMBER OF PAGES WHICH DO NOT
REPRODUCE LEGIBLY.**

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED			1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY N/A			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A			5. MONITORING ORGANIZATION REPORT NUMBER(S) AFWAL-TR-87-2036		
4. PERFORMING ORGANIZATION REPORT NUMBER(S)			7a. NAME OF MONITORING ORGANIZATION Aero Propulsion Laboratory Fuels and Lubrication Div. (AFWAL/POSF)		
6a. NAME OF PERFORMING ORGANIZATION STRAT Co.		6b. OFFICE SYMBOL (If applicable)	7b. ADDRESS (City, State, and Zip Code) AFWAL/POSF Wright-Patterson AFB OH 45433-6563		
6c. ADDRESS (City, State, and Zip Code) 4597 Jupiter Drive Salt Lake City UT 84124			9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER F33615-86-C-2660		
8a. NAME OF FUNDING/SPONSORING ORGANIZATION		8b. OFFICE SYMBOL (If applicable)	10. SOURCE OF FUNDING NUMBERS		
8c. ADDRESS (City, State, and Zip Code)			PROGRAM ELEMENT NO 65502F	PROJECT NO 3005	TASK NO 20
			WORK UNIT ACCESSION NO 63		
11. TITLE (Include Security Classification) Production of High Energy Aviation Fuels From Advanced Coal Liquids					
12. PERSONAL AUTHOR(S) John Downen					
13a. TYPE OF REPORT Phase I Final Report		13b. TIME COVERED FROM Jun 86 to Feb 87	14. DATE OF REPORT (Year, Month, Day) April 1987		15. PAGE COUNT 79
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP	AVIATION FUEL, HIGH ENERGY Coal Liquids, Hydrogenation of Coal Liquids, Advanced Fuels		
21	04				
07	01				
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The objective of this program was to assess the feasibility of producing advanced fuels from coal liquids. The coal liquids used in this program were produced from the Close Coupled Integrated Two Stage Liquifaction System (ISTL) plant at Wilsonville Alabama. The liquids were derived from subituminous coal, Wyodack upper seam, from Gillete Wyoming. The coal liquids were distilled into a 300-625°F fraction. Hydrogenation experiments were conducted on this distillate fraction using the Hydrogenation Reactor System (HRS) located at Wright-Patterson AFB, Ohio. Shell 424 nickel/molybdenum catalyst was used for heteroatom removal and aromatics saturation. The report details the analysis of these hydrogenated candidate fuels. <i>Key words:</i>					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED-UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED		
22a. NAME OF RESPONSIBLE INDIVIDUAL W. E. HARRISON			22b. TELEPHONE (Include Area Code) 513-255-6601		22c. OFFICE SYMBOL AFWAL/POSF

DD Form 1473, JUN 86

Previous editions are obsolete

SECURITY CLASSIFICATION OF THIS PAGE

UNCLASSIFIED

TABLE OF CONTENTS

INTRODUCTION AND ACKNOWLEDGMENTS.....	1
THE HYDROGENATION REACTION SYSTEM.....	3
TEST RESULTS.....	4
TEST DATA.....	7
TABLE 1 TEST MATRIX.....	7
TABLE 2 DATA ON COAL LIQUID FEEDSTOCK.....	8
TABLE 3 EFFECT OF REACTION TEMPERATURE ON PRODUCTS.....	8
TABLE 4 EFFECT OF PRESSURE ON PRODUCTS.....	9
TABLE 5 EFFECT OF RESIDENCE TIME ON PRODUCTS.....	9
TABLE 6 EFFECT OF CATALYST TYPE ON PRODUCTS.....	9
TABLE 7 EFFECT OF CATALYST TYPE ON PRODUCTS.....	10
TABLE 8 EFFECT OF CATALYST TYPE ON PRODUCTS.....	10
RESULTS OF FURTHER ANALYTICAL WORK.....	10
TABLE 9 GC/MS RESULTS ON SELECTED PRODUCTS.....	12
TABLE 10 EFFECT OF REACTION TEMPERATURE ON PRODUCT COMPOSITION.....	12
TABLE 11 PROPERTIES OF C ₁₀ HYDROCARBONS.....	13
CONCLUSIONS.....	15
RECOMMENDATIONS.....	15
REFERENCES.....	16
APPENDIX A GRAPHIC PRESENTATION OF TEST DATA.....	A1
FIGURE 1 EFFECT OF TEMPERATURE ON SULFUR.....	A1
FIGURE 2 EFFECT OF TEMPERATURE ON NITROGEN.....	A2
FIGURE 3 EFFECT OF TEMPERATURE ON HYDROGEN.....	A3
FIGURE 4 EFFECT OF TEMPERATURE ON DENSITY.....	A4
FIGURE 5 EFFECT OF PRESSURE ON SULFUR.....	A5
FIGURE 6 EFFECT OF PRESSURE ON NITROGEN.....	A6
FIGURE 7 EFFECT OF PRESSURE ON HYDROGEN.....	A7
FIGURE 8 EFFECT OF PRESSURE ON DENSITY.....	A8
FIGURE 9 EFFECT OF PRESSURE ON SULFUR.....	A9
FIGURE 10 EFFECT OF PRESSURE ON NITROGEN.....	A10
FIGURE 11 EFFECT OF PRESSURE ON HYDROGEN.....	A11
FIGURE 12 EFFECT OF PRESSURE ON DENSITY.....	A12
FIGURE 13 EFFECT OF CATALYST TYPE ON SULFUR.....	A13
FIGURE 14 EFFECT OF CATALYST TYPE ON NITROGEN.....	A14
FIGURE 15 EFFECT OF CATALYST TYPE ON HYDROGEN.....	A15
FIGURE 16 EFFECT OF CATALYST TYPE ON DENSITY.....	A16
FIGURE 17 EFFECT OF CATALYST TYPE ON SULFUR.....	A17
FIGURE 18 EFFECT OF CATALYST TYPE ON NITROGEN.....	A18
FIGURE 19 EFFECT OF CATALYST TYPE ON HYDROGEN.....	A19
FIGURE 20 EFFECT OF CATALYST TYPE ON DENSITY.....	A20
FIGURE 21 EFFECT OF CATALYST TYPE ON SULFUR.....	A21
FIGURE 22 EFFECT OF CATALYST TYPE ON NITROGEN.....	A22
FIGURE 23 EFFECT OF CATALYST TYPE ON HYDROGEN.....	A23
FIGURE 24 EFFECT OF CATALYST TYPE ON DENSITY.....	A24
FIGURE 25 EFFECT OF TEMPERATURE ON AROMATICS.....	A25
FIGURE 26 EFFECT OF TEMPERATURE ON NAPHTHENES.....	A26
FIGURE 27 EFFECT OF TEMPERATURE ON OLEFINS.....	A27
FIGURE 28 EFFECT OF TEMPERATURE ON PARAFFINS.....	A28
FIGURE 29 TEMPERATURE VS SATURATES & UNSATURATES.....	A29

APPENDIX B.....	B1
ANALYSIS OF COAL LIQUID.....	B1
RUN CONDITIONS AND SCHEMATIC OF THE TSL FACILITY.....	B5
LETTER AND REPORT FROM J & A ASSOCIATES, INC.....	B9
SCHEMATIC OF THE HRS.....	B19
BROCHURE ON THE SHELL 424 CATALYST.....	B20
DIAGRAM AND DISCUSSION OF REACTOR.....	B22
APPENDIX C.....	C1
TEST METHODS.....	C1
TEST DATA.....	C3

Accession For	
NTIS GRA&I	☒
DTIC TAB	☐
Unannounced	☐
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	



PRODUCTION OF HIGH ENERGY AVIATION FUELS

from

ADVANCED COAL LIQUIDS

John Downen, Principal Investigator

Strat Co., 4597 Jupiter Drive, Salt Lake City, Utah

March 15, 1987

Introduction and Acknowledgments

Strat Co. initiated an innovative research effort supported by the AFWAL/Aero Propulsion Laboratory to examine the feasibility of producing higher energy aviation fuels from domestic coal resources. This effort would address two issues; first, it would assess aspects of a domestic resource, coal liquids, that have heretofore been considered to be of little value as an aviation fuel feedstock; and, secondly, it would produce liquids suitable as fuels for aircraft of increased speed and range.

The work was conducted at the Fuels Branch of the Aero Propulsion Laboratories at Wright-Patterson Air Force Base. The assistance of the staff of the Fuels Branch is gratefully acknowledged, for its conduct of the experimental and analytical work of the project.

Conventional aviation fuels are comprised primarily of normal and iso-paraffins which are the major constituents of most crude oils. Aromatic molecules naturally exist in some crude oils and others are generated in refinery cracking and reforming processes. Olefins are almost exclusively a result of the cracking and coking process. Aromatic molecules are significantly more dense than paraffinic molecules and therefore contain substantially more energy per unit volume. Unfortunately, aromatics do not burn well in conventional engines and as a consequence maximum aromatics specifications and corresponding minimum smoke point specifications have been established for conventional jet fuels.

Naphthenic molecules appear to be the best prospects for improved hydrocarbon aircraft fuels. They retain most of the advantages of both paraffins and aromatics. Like paraffins, the naphthenes are saturated and thus are more stable than the unsaturated aromatics and olefins. Naphthenes also burn more like paraffins, without most of the smoke problems related to aromatics. Because naphthenes are cyclic paraffins, their density is greater than the normal and iso paraffins that make up most of the volume of

conventional fuels. Naphthenes therefore contain more energy per unit volume than conventional fuels.

Unfortunately, naphthenes do not naturally occur in most crudes in high enough concentrations to significantly affect the energy density of conventional aviation fuels. The most likely processes for making the volumes of naphthenes needed for advanced fuels will involve careful hydroprocessing of aromatic molecules of the appropriate size in order to saturate their double bonds without breaking their rings, thus converting them to naphthenes. Hydrocarbon liquids derived from certain coals contain high percentages of those aromatic molecules and may become one of the prime sources of the feedstocks needed for production of advanced fuels.

Coal tars as secondary products from coal gasification projects were originally considered as possible feedstocks for this program. As a first approximation, the characteristics of these materials were found to be not suitable for volume production of high quality fuels. The difficulties related to their processing, transport and storage and the potential carcinogenicity of the heavier tars were the principal factors in this decision. These materials also are now not expected to become widely available on the domestic market in the near future. For a number of reasons, therefore, a middle distillate of the coal liquids from the DOE/EPRI Advanced Two-Stage Liquifaction Facility (TSL) which is operated by Catalytic Inc. at Wilsonville, Alabama was determined to be a better feedstock for Phase I of this program.

The feedstock evaluated in this effort was derived from upper seam Wyodak (Wyoming subbituminous) coal which was processed at the TSL. It was produced in Run #251 during August 1986 under the sponsorship of the Department of Energy, the Electric Power Research Institute (EPRI) and several industrial supporters. A schematic of the TSL facility is included in Appendix B as is a report on the composition of the liquid before it was fractionated.

Two drums of the TSL liquids were fractionated by J&A Associates, Inc. of Golden, Colorado in a 25 gallon batch still. Only the 340°F to 590°F cut (ASTM D86) was shipped to AFWAL for hydrotreatment into candidate fuels (see Table 2). Appendix B also includes a letter report from J&A outlining the results of the distillation of the two drums along with pertinent data.

Sullivan^{1,2,3} had previously evaluated coal liquids from the TSL and produced fuels which met current jet fuel specifications. In this effort, the objective was not necessarily to produce specification fuels but to produce a variety of liquids which might be considered for future advanced applications. Because of a lack of product fractionation equipment and of fuel "polishing" facilities such as clay treatment and additives, no real attempt was made to produce finished specification fuels.

The effect of reaction conditions on the quality of fuel was determined within the limitations of the analysis equipment at AFVAL. In addition, this effort was Phase I of a more comprehensive program which would optimize fuel production conditions and evaluate these materials in simulated advanced propulsion applications. In the Phase II expansion of this effort, feedstocks other than coal liquids, including energetic gases, would also be evaluated and compared. Some details of the Phase II effort will be further discussed in the "Recommendations" Section of this report.

The Hydrogenation Reaction System

The reaction system used in this program was located in the Fuels and Lubrication Building (Building 490) in Area B at Wright-Patterson Air Force Base near Dayton, Ohio. The Hydrogenation Reaction System (HRS) was an automated pilot plant capable of studying the processing associated with feedstocks such as petroleum, heavy oils, tars, coal liquids, shale oil etc. Its main function, however, is to study hydrogenation and hydrocracking processes needed to produce suitable candidates for transportation fuels such as jet and diesel from a variety of sources. The unit was built by Xytel Inc. of Chicago from a design based on Air Force requirements.

Appendix B includes a schematic of the HRS. It is basically a two-reactor system with the capability of operating the reactors in series. For instance, in upgrading high fuel-bound nitrogen feed like western shale oil, the first reactor could be used as a "guard" reactor to remove nitrogen to the parts per million range, remove metals such as arsenic and iron and to saturate olefinic hydrocarbons, to keep the main hydrocracking reactor from being poisoned. This procedure is analogous to typical refinery operations with these materials and is used in the Unocal shale oil upgrading facility near Parachute, Colorado. However, in this study only one reactor was used since "life studies" on the catalyst were beyond the scope of the effort.

The HRS facility has the normal capability of supplying liquid and gaseous fluids to the reactors. The temperatures in the reactor are monitored and controlled by a thermocouple located in a thermowell on the centerline of the reactor. The thermocouple can traverse the length of the reactor. The facility has the capability of liquid feed rates up to 600 cc/hr with hydrogen flows being limited to approximately 35 standard cubic feet per hour. The reaction temperatures are limited to approximately 950°F with a pressure capability of nearly 3000psig. The reaction products can be scrubbed to remove hydrogen sulfide and ammonia and the gaseous products can be separated from the liquids. Gas analysis using an on-stream gas chromatograph can be made to facilitate accurate material balances. The facility is physically located in Room 154 of Building 490 in a high-bay

area equipped with more than adequate safety precautions. A complete description of the HRS is expected to be available in reports originating from the AFWAL Fuels and Lubrication Division and will not be presented here.

This facility will be of great value to the USAF as it develops its understanding of the processes for producing high quality fuels for future hypersonic aircraft. In addition the basic system is being modified so it can be configured to evaluate catalyst systems for endothermic fuels needed for high speed flight in the atmosphere. The operations made possible by this configuration will become the focus of the Phase II part of this program.

Test Results

A series of hydrogenation tests were performed on the coal liquid (340°-590° F) distillate to evaluate the effect of the reaction parameters on the resulting products. Sufficient products were obtained to provide analytical data to ascertain the quality of the products as suitable feedstock for candidate fuels. Complete jet fuel specifications on the products were not run since no attempt was made to produce "polished" usable fuels. Such tests as corrosivity and thermal stability could be met through correct finishing processes such as stripping and clay treatment. The object of this study was to produce suitable material for further refining including desired fractionation.

No attempt was made to evaluate catalyst life. Before a valid economic analysis of the cost of the products could be made, data on the life of the catalysts would have to be obtained by further work, as it has a major effect on refinery economics and must be maximized for production operations. Further work would also be needed in order to develop information for projections for scale-up and continuous production of any of these liquids. This was outside the scope of the Phase I study and will be developed from the Phase II data.

For this effort two catalysts were selected to represent conventional off-the-shelf hydrotreating catalysts. They were a nickle-molybdenum (NiMo) and a precious metal catalyst which usually has increased hydrogenation activity. Shell 424, NiMo on alumina, was tested on the feedstock in 85% of the runs and this data was compared to an Engelhard sulfided 0.5 wt% Pt on carbon catalyst which was acquired from the Hercules Research Center at Wilmington, DE. A descriptive brochure on the Shell 424 catalyst is included in Appendix B.

Reaction conditions studied included temperatures: 600°-700° F, pressures: 2000-2500 psig, and feedrates: 0.19-0.39 lbm/hr (LHSV: 0.5 to 1.0). Table 1 is the test matrix as the project was conducted. Further tests were scheduled with the Pt catalyst and another series was planned with a nickle-zinc catalyst but

strong symptoms of an imminent failure of the main hydrogen pump of the HRS ended the test series before they were run.

A summary description of the feedstock is presented as Table 2. Appendix B includes the reports from J&A associates and from Catalytic Inc. that describe it in more detail. Appendix B also includes a diagram of the reactor and a discussion of how it was loaded. Appendix A is a suite of graphic presentations of the test data.

The products were analyzed for density, weight percents of sulfur, nitrogen and hydrogen and for chemical types by GC/MS for certain key products. The data resulting from the sulfur analysis is anomalous. A moderate drop in sulfur concentration was observed as the feedstock was hydrotreated. However, under the severity of the operating conditions, it was expected that the sulfur would be essentially removed during hydrotreating. In spite of this problem, the data from the sulfur analyses are presented in this report. It is likely that the actual concentration of sulfur in the product is near zero. A more appropriate method of analysis for low sulfur concentrations will be used in Phase II.

A complete description of reaction conditions is included as Appendix C together with all test results except chromatographs and peak identification data.

Tables 3-8 describe the effects of reaction conditions on the products from the tests performed in this project. This effort, (SBIR Phase I), was to be a scoping study to indicate directionally where Phase II should be initiated to gain fuller insight into advanced fuel production from the point of view of reaction conditions and economics.

Most of the reactions studied were carried out over the Shell 424 catalyst. The effect of reaction temperature at 2000psig is presented in Table 3. The products produced at 600°-700°F are compared to the feedstock. At a LHSV of 1 and hydrogen feed of 12,100 scf/bbl, sulfur and nitrogen contents were reduced while the hydrogen content of the products increased with temperature. The density of the products decreased (increased API gravity) due primarily to the hydrogenation. Very little cracking occurred at these conditions but probably would have become significant at higher reactor temperatures and lower pressures. The apparent sulfur reduction with temperature was not dramatic (from 400 to 300ppm) and is almost certainly indicative of imprecise analytical techniques. The feedstock sulfur content (438ppm) is higher than the J&A analyses (131ppm) indicated. This anomaly will be cleared up in Phase II with a better understanding of these analytical methods.

Nitrogen levels were reduced from 3000ppm to 20-60ppm at the test temperatures; these figures are probably more accurate than the sulfur assays. Assuming that the data in Table 3 is accurate,

then the Shell 424 catalyst is not effective in producing a sulfur-free or nitrogen-free product for advanced fuel applications where these components would cause high temperature instabilities, although further work and better analyses may lead to conditions of better performance by the catalyst. In any event, the Shell 424 catalyst could be used in guard reactors to reduce nitrogen levels and perhaps trace metal and olefin contents to levels where extremely active catalysts would not be poisoned in downstream hydrogenation/hydrocracking operations.

In Table 4 the effect of reaction pressure on hydrogenation was evaluated in several of the tests. The effect of increased pressure on the reduction of sulfur and nitrogen and the increase in hydrogen content are not apparent from tests run at 2000psig and 2500psig. Tests at lower and higher pressures were beyond the scope of this effort and probably would have given more insight into the effect of greater pressure range. The tests in Table 4 were all run at 700°F, LHSV=1 and using the Shell 424 catalyst. From this minimal data there would seem to be no need to hydrotreat at higher pressures.

The effect of residence time was evaluated and presented in Table 5. These tests were performed at LHSV of 1 and 0.5, at a pressure of 2500psig and temperature of 700°F using the Shell 424 catalyst. Here again, sulfur content of the product was apparently not greatly influenced by the greater residence time (lower flow rate). The nitrogen content was reduced from 74ppm to 50ppm at the lower flow rate and the hydrogen content increased by 2% to 12.93%

Table 6 evaluates the effect of the Shell 424 catalyst compared with the presulfided Engelhard 0.5 wt% Pt on carbon and clearly indicates, with the minimum data obtained, that the Pt is not as effective as the NiMo in removing sulfur and nitrogen from the feed. Also the hydrogen content of the Pt catalyst-produced product was lower than that with the Shell 424 material. These bracketed tests were run at reaction temperature of 600°F, pressure of 2000psig and LHSV=1.

The effect of a higher reaction temperature, 700°F, was demonstrated on the two catalyst types in Table 7. The same results are found as those observed at the lower reaction temperatures. At 700°F the Pt catalyst also produced a much higher gravity (41.70° API) indicating that some cracking may have been occurring. It is highly likely that such trace compounds as sulfur, nitrogen and metals may have poisoned the usually highly reactive Pt catalyst.

In Phase II the Pt catalyst will be used to evaluate advanced fuel production but an appropriate guard reactor will be used. As Pt catalysts are expected to function well in an endothermic regime, the data from the Pt catalyst in this program may serve as baseline information for the next phase. In Phase I, this

approach was not practical based on scheduling and funding limitations.

Results on the two catalyst types at lower feedrates are presented in Table 8 and the results and conclusions are similar to those in Table 7.

Test Data

TABLE 1

TEST MATRIX

test no.	temp. °F	catalyst type	press. psig	feedrate lbs mass/hr	LHSV	gas rate scfh **
HRS-016	FEEDSTOCK					
HRS-017	600	NI-MO	2000	0.39	1	15
HRS-018	600	NI-MO	2000	0.39	1	15
HRS-019	600	NI-MO	2000	0.39	1	15
HRS-020	650	NI-MO	2000	0.39	1	15
HRS-021	695	NI-MO	2000	0.39	1	15
HRS-022	FEEDSTOCK					
HRS-023	700	NI-MO	2000	0.39	1	15
HRS-024	700	NI-MO	2000	0.39	1	15
HRS-025	700	NI-MO	2500	0.39	1	15
HRS-026	700	NI-MO	2500	0.39	1	15
HRS-027	700	NI-MO	2500	0.39	1	15
HRS-028	700	NI-MO	2500	0.19	0.5	15
HRS-029	700	NI-MO	2500	0.39	1	15
HRS-030	700	NI-MO	2500	0.19	0.5	15
HRS-031	600	NI-MO	2000	0.39	1	15
HRS-032	600	NI-MO	2000	0.39	1	15
HRS-033	600	NI-MO	2000	0.39	1	15
HRS-034	600	Pt	2000	0.39	1	15
HRS-035	700	Pt	2000	0.39	1	15
HRS-036	700	Pt	2000	0.19	0.5	15

** NOTE: This converts to 12,096 (12,100) scf/bbl of feedstock at LHSV of 1, and 24,200 scf/bbl at LHSV of 0.5, on a one pass through basis (no hydrogen is recycled).

TABLE 2

DATA ON COAL LIQUID FEEDSTOCK
(From J & A Associates Report)

Quantity: 61.3 gallons (at 60 degrees F)
Volume per unit of liquid: 58.4

Elemental Analysis, wt%

Carbon	86.83
Hydrogen	12.84
Nitrogen	0.25
Sulfur	0.01

Distillation (ASTM-D86)

°F

1BP	340
10% recovered	382
30% recovered	437
50% recovered	486
70% recovered	524
90% recovered	566
end point	590

TABLE 3

EFFECT OF REACTION TEMPERATURE ON PRODUCTS

Catalyst: Shell 424(Ni/Mo) Feed Rate: 0.39 lbm/hr
Pressure: 2000 psig H2 Feed: 12,100 scf/bbl
LHSV: 1

Analyses

Reaction Temperature
°F

	Sulfur wt %	Nitrogen wt %	Hydrogen wt %	Gravity API
feed	0.044	0.300	12.01	27.45
600	0.041	0.070	12.44	29.77
650	0.033	0.002	12.93	31.61
695		0.006	12.76	31.27
700	0.034	0.006	12.78	31.27

TABLE 4

EFFECT OF PRESSURE ON PRODUCTS

Catalyst: Shell 424 Feed Rate: 0.39 lbm/hr
 Temperature: 700°F LHSV: 1

Reactor Pressure psig	Analyses			
	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	0.300	12.01	27.45
2000	0.034	0.006	12.78	31.27
2500	0.036	0.007	12.73	31.24

TABLE 5

EFFECT OF RESIDENCE TIME ON PRODUCTS

Catalyst: Shell 424 Temperature: 700°F
 Pressure: 2500 psig

Feed Rate lbm/hr (LHSV)	Analyses			
	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	0.300	12.01	27.45
0.39 (1)	0.032	0.0074	12.70	31.14
0.19 (0.5)	0.029	0.0050	12.93	32.03

TABLE 6

EFFECT OF CATALYST TYPE ON PRODUCTS

Pressure: 2000 psig
 Temperature: 600°F
 Feedrate: 0.39 lbm/hr
 LHSV: 1

Catalyst Type	Analyses			
	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	0.300	12.01	27.45
Shell 424 (Ni/Mo)	0.041	0.070	12.44	29.77
Englehard (0.5 Pt)	0.040	0.262	12.29	28.53

TABLE 7

EFFECT OF CATALYST TYPE ON PRODUCTS

Pressure: 2000 psig
 Temperature: 700° F
 Feedrate: 0.39 lbm/hr
 LHSV: 1

Catalyst Type	Analyses			
	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	0.300	12.01	27.45
Shell 424 (Ni/Mo)	0.034	0.006	12.78	31.27
Englehard (0.5%Pt)	0.038	0.161	12.57	41.78

TABLE 8

EFFECT OF CATALYST TYPE ON PRODUCTS

Pressure: 2000 psig
 Temperature: 700° F
 Feedrate: 0.19 lbm/hr
 LHSV: 0.5

Catalyst Type	Analyses			
	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	0.300	12.01	27.45
Shell 424 (Ni/Mo)	0.029	0.005	12.93	32.03
Engelhard (0.5% pt)	0.040	0.151	12.49	29.74

Results of Further Analytical Work

For selected hydrotreating tests, gas chromatographic/mass spectographic analyses were run. This data is useful in identifying basic hydrocarbon types. Saturated hydrocarbons are either straight-chained normal and iso paraffins or mono- and dicycloparraffins. These types of structures represent the highest hydrogen content possible, with n-paraffins having the highest possible content for a given carbon number. The most stable unsaturated hydrocarbons are aromatics, here represented by alkylbenzenes, indans and tetralins, and naphthalenes. These materials are deficient of hydrogen atoms but are stable due to resonance within the aromatic ring. The cyclic compounds can be hydrogenated to cyclic paraffins, i.e. naphthalenes become dicycloparraffins with the saturation of all carbon atoms.

In this effort, the GC/MS data were obtained on selected products and compared to the feedstock. This data is presented in Table 9. Seven classes of compounds are quantified. The first three categories are paraffinic (completely saturated) while the last four are unsaturated aromatics and olefins. Olefins are unsaturated hydrocarbons which are not stabilized by resonance, are usually identified as being unstable and are characterized as gum formers, depending on type. Di-olefins are usually the most unstable.

The coal liquid from Wilsonville was an excellent potential refinery feedstock. It was hydrogenated to a fairly high degree in its production as its hydrogen content was 12.01 wt% with only 25.3% aromatics and 1.2 wt% olefins. This data is summarized by general group (paraffins, naphthenes, aromatics and olefins). Table 10 shows the effect of reaction conditions on these hydrocarbon types. Using the Shell 424 catalyst and reaction pressure of 2000 psig and liquid feedrate of 0.39 lb (mass) / hour (LHSV=1) the effect of increasing reaction temperature is presented. The paraffin content dropped slightly from 16.1% to 13-14% as the temperature was increased. The naphthenes, which are the candidate compounds for use as high density fuels, increased with temperature commensurate with the decrease of aromatics. Also, as would be expected, the total hydrogen content of the product increased as the aromatics and olefins became saturated. As its hydrogen content approaches 13%, a fuel becomes more acceptable for contemporary jet engines. Future engines will benefit from the increased volumetric energy by being designed to operate efficiently on lower hydrogen content and heavier cyclic fuels.

The high remaining paraffin content is probably representative of the long chained waxes which are typical of liquids from subbituminous coal, which have higher freeze points and which would tend to raise the freeze point of the products. The freeze points of these products are significantly higher than those of most conventional fuels. Not much cracking occurred under these test conditions. The freeze points could be decreased by dewaxing or isomerization of the products although a less expensive procedure may be just to decrease the end point by fractionation. Another option would be to use a higher rank coal as the liquifier feedstock which would produce a higher percentage of aromatics and less paraffins. The fractionation facilities were not operable at the Air Force laboratory for Phase I but should be available in Phase II to facilitate production of as broad a range as possible of suitable materials. Process economics will be compared based on using wider cuts and converting the waxes to lower melting isomers versus fractionating a more narrow boiling range product.

Cyclohydrocarbons are more dense than the corresponding paraffins, see Table 11 where n-decane is compared to decahydronaphthalene (decalin). Both molecules contain ten carbon atoms. The specific gravity of the straight chained n-

decane is 0.7342 compared to 0.9011 for the cis-decalin. Also the dicyclic decalin produces a lower freeze point and an 18% increase in volumetric heating value. The drawback in using cyclic hydrocarbons is the reduction in hydrogen content which affects fuel combustibility in terms of increased flame radiation and soot formation. These shortcomings can be dealt with and to a considerable extent overcome by engine design.

Table 9

GC/MS RESULTS ON SELECTED PRODUCTS

Test Number, HRS-
(See Table 1 For Corresponding HRS Test Operating Conditions)

	016	018	020	021	024	028	031	034
Hydrocarbon type, wt%								
Paraffins	16.1	13.3	13.4	13.2	14.0	12.1	15.0	13.9
Monocycloparaffins	33.4	37.0	38.0	38.0	39.1	39.6	34.9	33.8
Dicycloparaffins	24.0	26.6	33.7	32.2	30.6	35.3	25.3	27.5
Alkylbenzenes	7.1	6.3	5.5	5.9	5.8	5.2	6.4	6.8
Indan & Tetralins	13.9	13.1	7.4	8.6	8.3	6.0	13.4	14.0
Naphthalenes	4.3	3.1	1.5	1.6	1.8	1.1	3.4	3.1
Olefins	1.2	0.7	0.5	0.5	0.5	0.6	0.6	0.9

Table 10

EFFECT OF REACTION TEMPERATURE ON PRODUCT COMPOSITION

Catalyst: Shell 424
Pressure: 2000 psig
Feed Rate: 0.39 lbm/hr
H2 Feed: 12,100 scf/bbl

Hydrocarbon type, wt %	Reaction Temperature, ° F				
	feedstock	600	650	695	700
Paraffins	16.1	13.3	13.4	13.2	14.0
Naphthenes	57.4	63.6	71.7	70.2	69.7
Aromatics	25.3	22.5	14.4	16.1	15.9
Olefins	1.2	0.7	0.5	0.5	0.5
Total saturates	73.5	76.9	85.1	83.4	83.7
Total unsaturates	26.5	23.2	14.9	16.6	16.4
Hydrogen, wt%	12.01	12.40	12.93	12.76	12.93

Table 11

PROPERTIES OF C₁₀ HYDROCARBONS

	n-decane	decalin
Formula	C ₁₀ H ₂₂	C ₁₀ H ₁₈
Molecular Weight	142.29	138.25
Specific Gravity	0.7342	0.9011
Boiling Point, °F	345.5	384.5
Freeze Point, °F	-21.4	-45.6
Characterization Factor, K	12.61	10.50
Hydrogen, Wt%	15.59	13.12
Net Heat Of Combustion		
BTU/lb	19,017	18,324
BTU/gal	116,410	137,660

Conclusions

Although the US interest in alternative domestic fossil fuel resources is currently in a lull, it is of interest to visualize the potential for producing from them not only high quality conventional fuels but the families of advanced fuels needed for the next generations of aircraft. Future effort in this area must be timely, aggressive, meaningful and technically sound.

Comparisons of characteristics of products made with current fuel specifications⁴ indicates that the Strat Co. effort demonstrated, within the constraints of the SBIR Phase I resources, that coal liquids could be processed into quality fuels. The feedstock which was fractionated into a cut suitable for jet fuel production was a high grade material; as received, the concentrations in it of sulfur, nitrogen and oxygen were lower than in most petroleum crudes. This material had been hydrostabilized as part of its production. Liquids from the proper coals processed in this facility undoubtedly represent one of the best sources currently available for feedstocks for experimental high density jet fuels. Developing the concept of endothermic or heat sink fuels and the need for cyclic hydrocarbons, like those available from liquified coal, could provide an impetus not only for initiating a viable domestic coal liquids industry but for producing quality advanced fuel for 21st century fighters, interceptors, cruise missiles and aerospace planes and ultimately for production of refinery feedstocks to supplement conventional crudes.

Using conventional processing technology and limited support, Strat Co. demonstrated that advanced fuels are possible within the state-of-the-art. The Shell 424 catalyst is representative of this technology and produced suitable feedstock for future fuels. More extensive information is needed, however, to fully

define future fuel candidates and areas where more sophisticated processing is needed. Pure or nearly pure hydrocarbon compounds or mixtures of them will probably be needed for certain advanced missions; these materials must be identified and specified and arrangements made for their production in a timely and economical manner.

Future efforts and modifications to the hydrotreating facility should enable the AF to study basic heat effects on fuels to gain insight into the realistic reaction kinetics and potential heat sink capabilities of candidate fuels. This information would then be used to design, build and operate a simulator for completely evaluating the fuel/fuel systems interface under more realistic conditions.

In looking at the applications of this data to further study for the production of advanced fuels from coal liquids, the hydrocarbon species needed for endothermic fuels, high density fuels and the other advanced concepts are most likely to be naphthene molecules that are fully saturated and that don't have the problems associated with fuels containing high percentages of aromatics. From the data it is clear that we have substantially increased the percentage of naphthenes. Most coal liquids are very rich in aromatics and most of the aromatics can be converted to naphthenes by proper hydrotreating. Obviously, most of the double bonds are saturated to form naphthenes although some of the rings are broken and the aromatics in that case are converted to alkanes.

Under the operating conditions of this study, the aromatics percentage of the treated product was higher than for specification fuels. By further variations of the operating parameters and catalyst selection this liquid could certainly be made into specification fuel. In addition, this treatment would also increase the concentration of naphthenes in the liquid.

The relative concentrations of the various hydrocarbon species vary greatly among coals of different origin, quality, and rank. For start material, one would select a high volatile, low-ash, low sulfur coal of medium rank. The details of the operating parameters of the coal-to-liquid conversion unit will also affect the relative concentrations of the hydrocarbon species formed. These processes can typically be varied to affect, within limits, the amount of aromatics produced from any particular coal; hence the selection process for producing any particular advanced fuel should commence with coal selection, followed by coal-to-liquid process selection and optimization of process operating parameters, then proceed through selection of a sequence of processes and corresponding operating parameters to distill, hydrotreat, and further fractionate and concentrate the desired species. Final concentration would probably be accomplished mainly by liquid-liquid extraction techniques.

Initially it would appear that producing an advanced fuel by selecting an optimum coal that is rich in aromatics , producing liquids from it under the proper conditions, hydrotreating the aromatics to naphthenes and concentrating them may furnish advanced fuels at lower cost than making them by synthesis from pure compounds. It also is likely that, although a group of similar naphthenic compounds would be produced rather than single compounds, the desired fuel characteristics that are being determined from work with single compounds could be obtained from such a group.

After hydrotreating there will still be some aromatics and paraffins remaining in the liquids. There are a number of commercial processes available to remove both paraffins and aromatics to arrive at a liquid that is composed entirely of naphthenic compounds of similar molecular weight. Following are representative examples:

For extracting the aromatics, one of a number of commercial liquid-liquid extraction processes could be used. Conceptually, this involves a solvent that dissolves the aromatics but not the paraffins or naphthenes. Commercial processes of this type are sulfolane extractors used to remove benzene, toluene, and xylene from high-octane reformat, NMP (n-methyl pyrrolidine) used to remove aromatics from lube oils, and SO₂ extraction used to remove aromatics from jet fuel. These are the obvious processes and there are others that could be adapted to this application.

A number of commercial processes exist for eliminating paraffins. Molecular sieves are available designed so that naphthenes and probably much of the iso-paraffins will pass through the sieve and the paraffins will not, and therefore can be recovered by back-flushing. In addition, there are two common commercial dewaxing catalysts, one licensed by British Petroleum which selectively breaks the long chains into light ends under hydrotreating conditions and lowers the pour point (or freeze point) of the liquid. The other, licensed by Mobil, behaves much like a fluid catalytic cracking unit with a hydrogen atmosphere where the paraffins are generally converted to olefins and aromatics in almost a de-hydrogenation reaction. A third catalytic dewaxing process was developed by Union Oil Co. in the late 1970s for dewaxing hydrotreated shale oil to reduce its pour point so it could be pipelined from Parachute Creek to the Fruitita refinery without the necessity for heating the pipeline. This was not commercially available as of two or three years ago, but arrangements could probably now be made for it.

Recommendations

The AF Fuels Branch has prided itself in keeping pace with fuel development activities. This became clear with the leadership role it took in developing fuels from domestic (western) oil shale. However, recent cutbacks in energy research by both industry and government dictate that the AF play a larger role if advanced fuels are ever to be practical. Recent funding cuts and manpower reductions require assessment of the modus operandi of the Laboratory. Research contracts must be well defined and hard hitting; opening up new areas of advancement and not merely "reploughing old turf".

Strat Co. plans to assist the AF in this challenging new endeavor. For Phase II, an aggressive, imaginative program will be proposed to assist in upgrading the in-house capability of the AF Fuels Laboratory. Outside consultants from industry and academia will be made available to review the background of fuel requirements and areas of future development. The internal facilities of the AF will be operated in an efficient manner to develop prototype fuels in a timely fashion. Current equipment should be modified to supply basic heat sink and chemical reaction parameters for future designs. Chemical analyses must be improved in both type and precision. Difficulty in obtaining correct data in a timely fashion will not be satisfactory if the AF is to move out in this area.

Strat Co. will present a proposal for a Phase II program which will enable the AF to begin to answer the major fuels-related questions posed by Project Forecast II before they disappear by default from lack of attention.

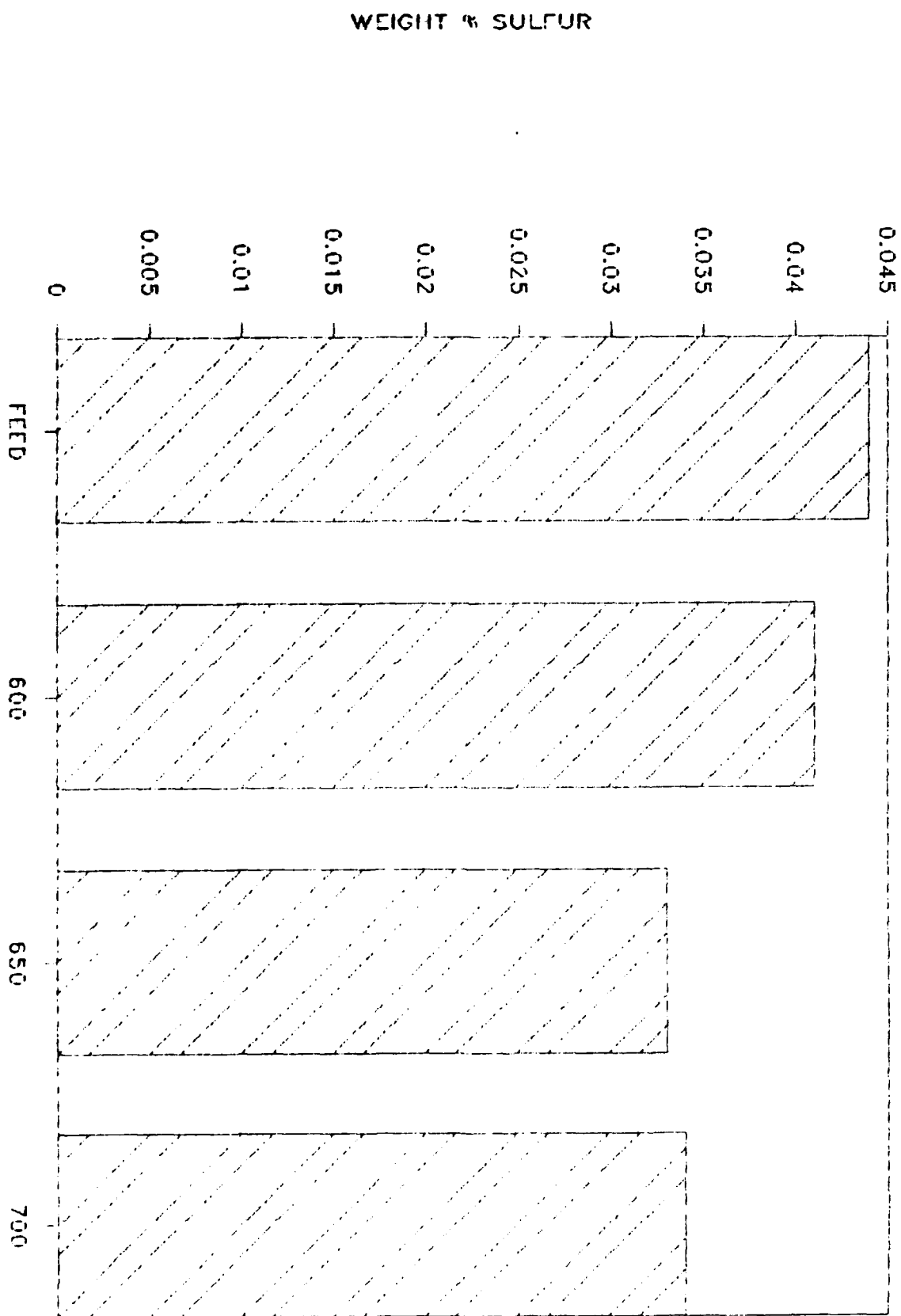
References

1. Richard F. Sullivan, "Two-Stage Catalytic Hydrocracking of ITSL Oil for Jet Fuel and Naphtha", Symposium on Catalytic Processes in Coal Conversion, presented before the Division of Petroleum Chemistry, Inc., American Chemical Society, Chicago Meeting, September 8-13, 1985
2. Richard F. Sullivan, "Effects of Feed Boiling Range on Hydrotreating of Wyodak and Illinois ITSL Oils", Paper for DOE Direct Liquefaction Contractor's Conference, Pittsburgh, Pennsylvania, November 19-21, 1985
3. Richard F. Sullivan and Harry A. Frumkin, "Refining Coal Liquids: Where We Stand" Chevron Research Company, undated
4. Coordinating Research Council, Inc. "Handbook of Aviation Fuel Properties" 1983, CRC Report No. 530, CRC, Inc. 219 Perimeter Center Parkway, Atlanta GA 30346

A P P E N D I X A
GRAPHIC PRESENTATION OF TEST DATA

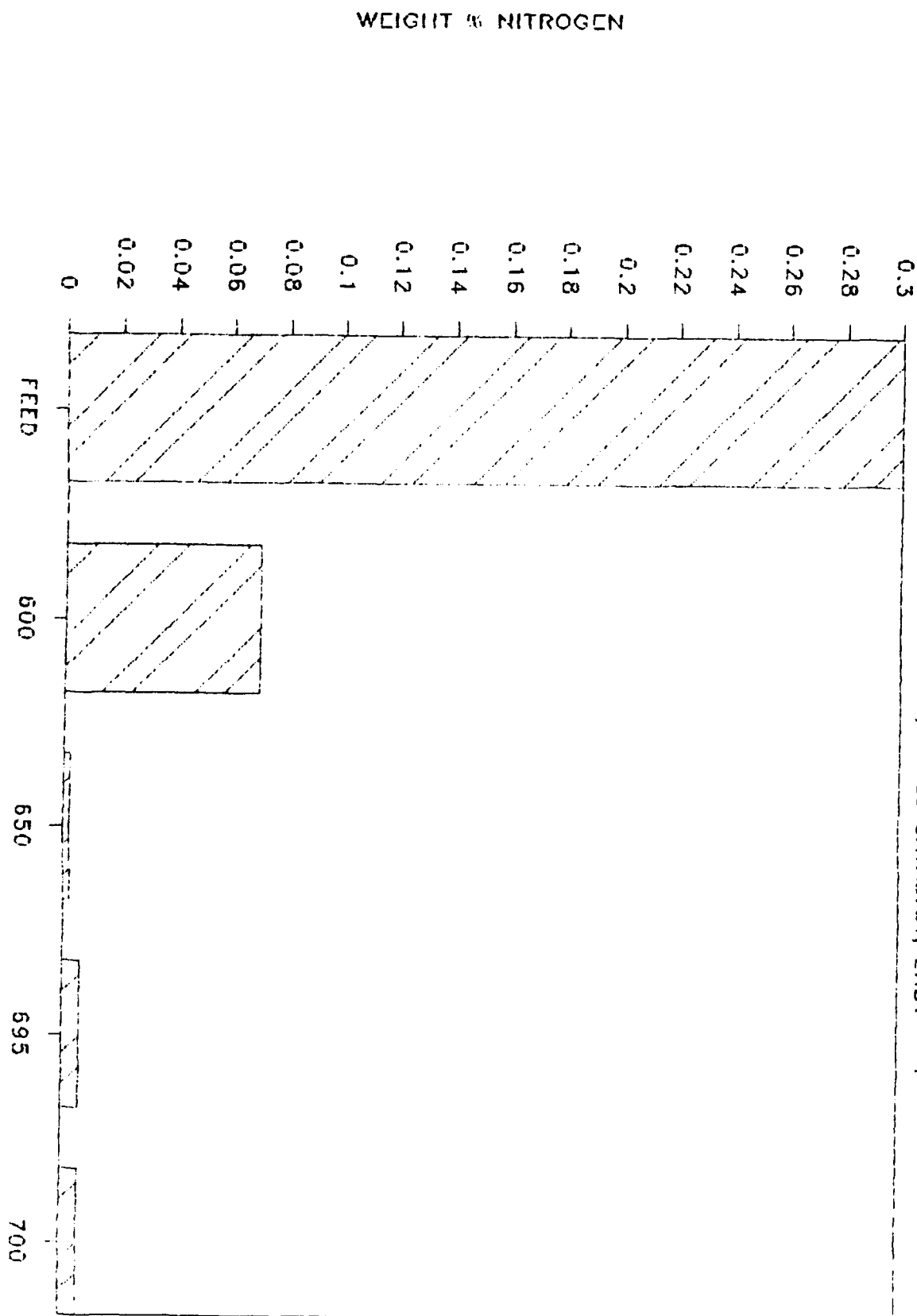
EFFECT OF TEMPERATURE ON SULFUR

2000 PSIG, SHELL CATALYST, LHSV = 1



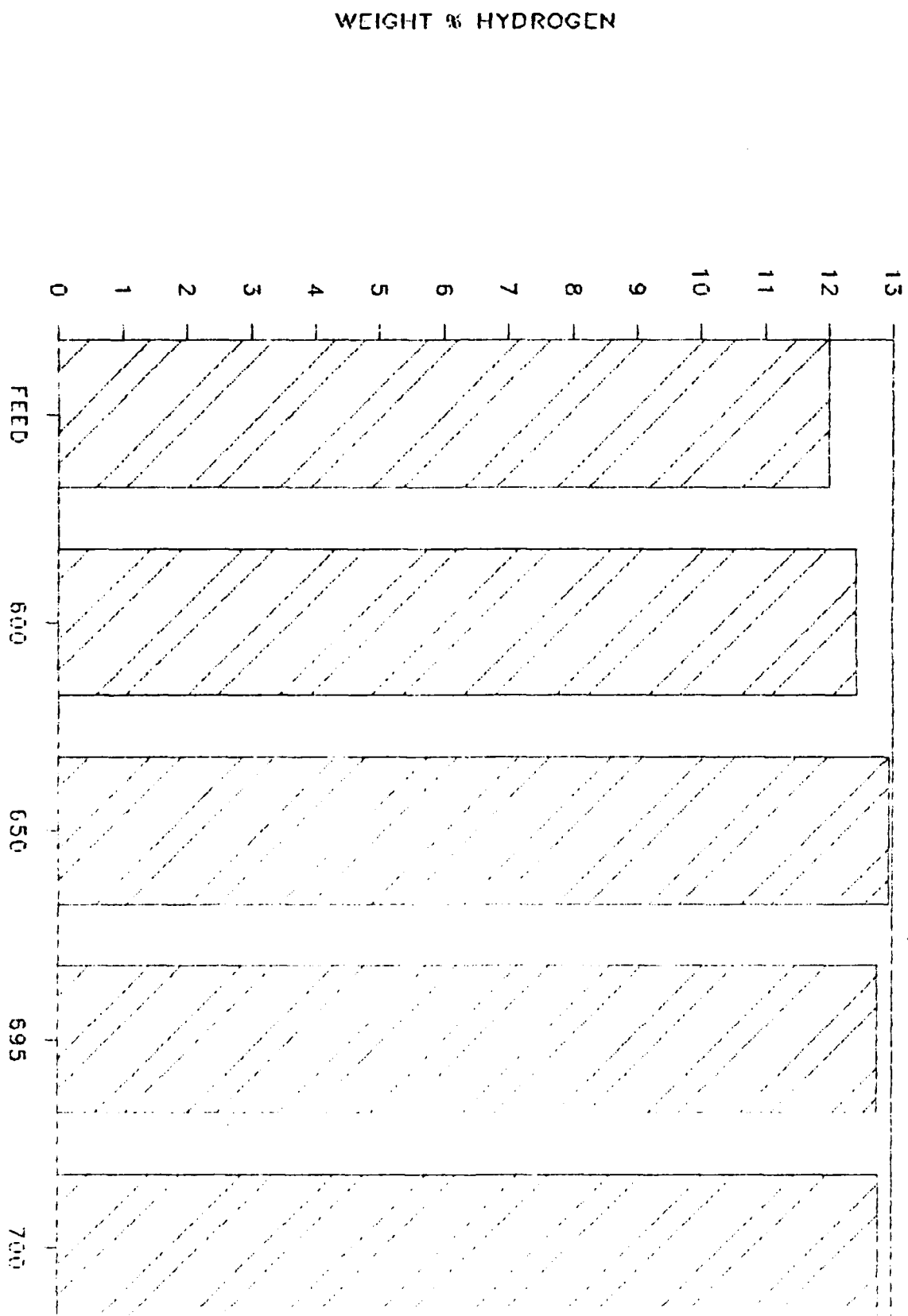
EFFECT OF TEMPERATURE ON NITROGEN

2000 PSIG, SHELL CATALYST, LHSV = 1



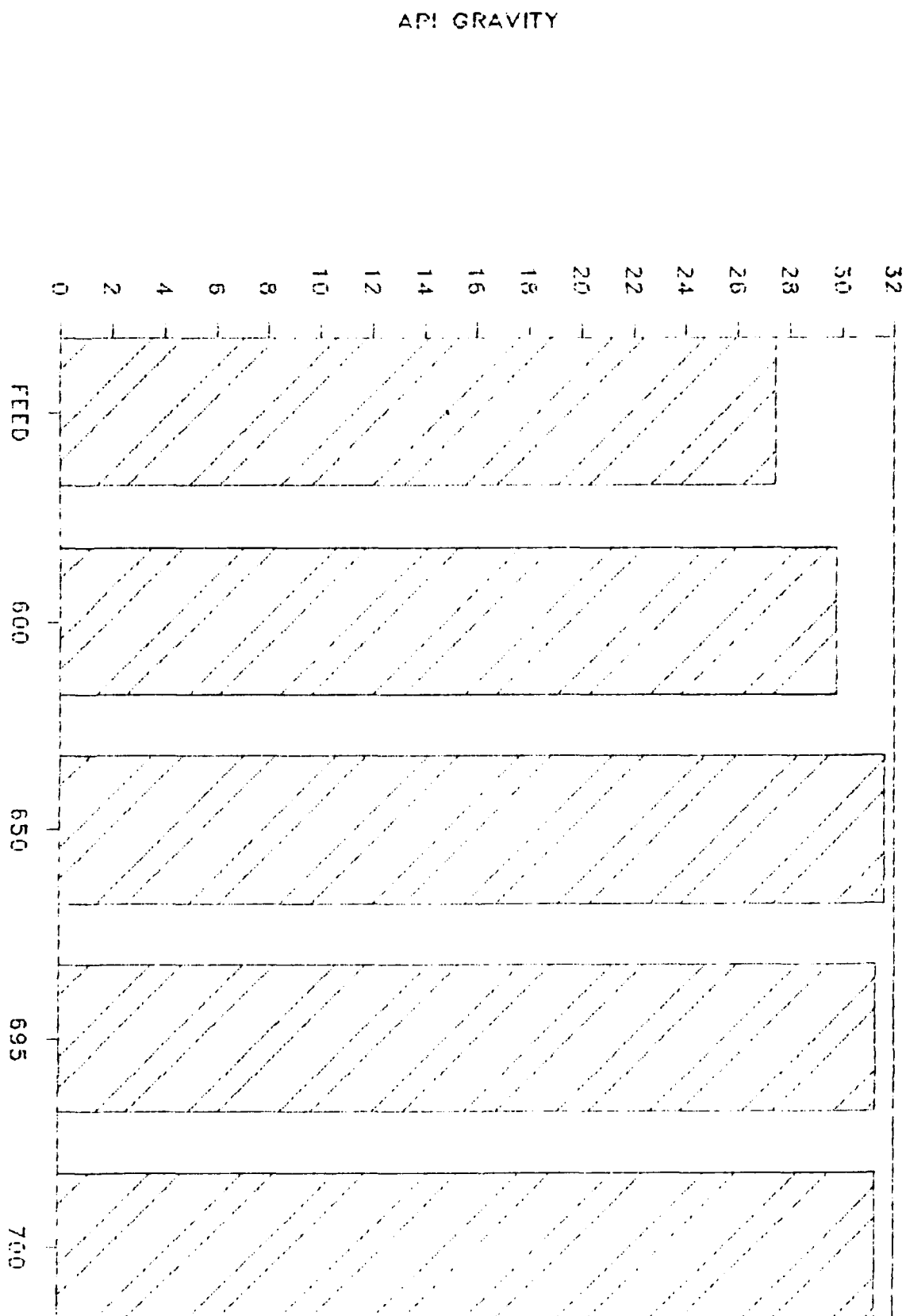
EFFECT OF TEMPERATURE ON HYDROGEN

2000 PSIG, SHELL CATALYST, LHSV = 1



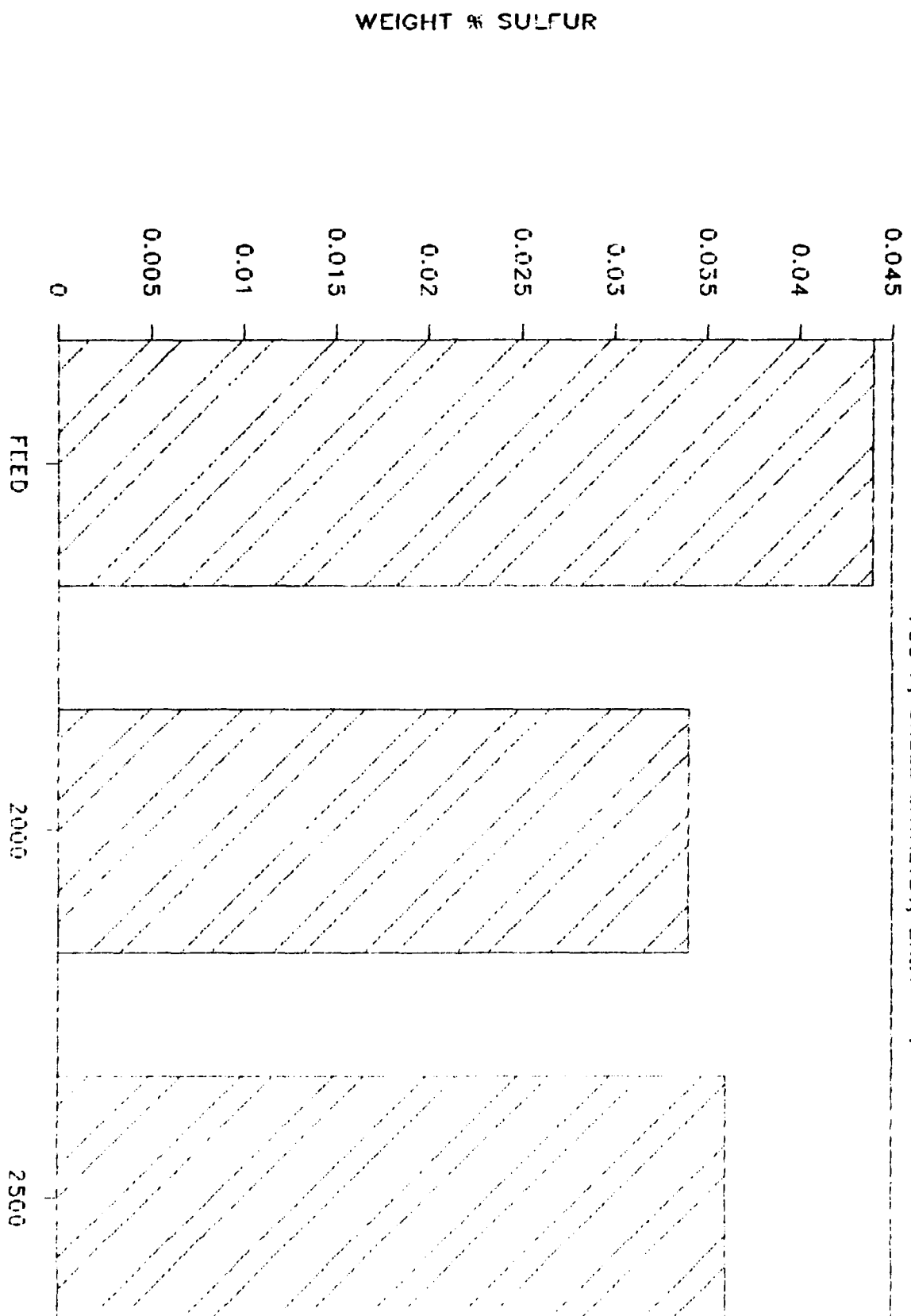
EFFECT OF TEMPERATURE ON DENSITY

2000 PSIG, SHELL CATALYST, LHSV = 1



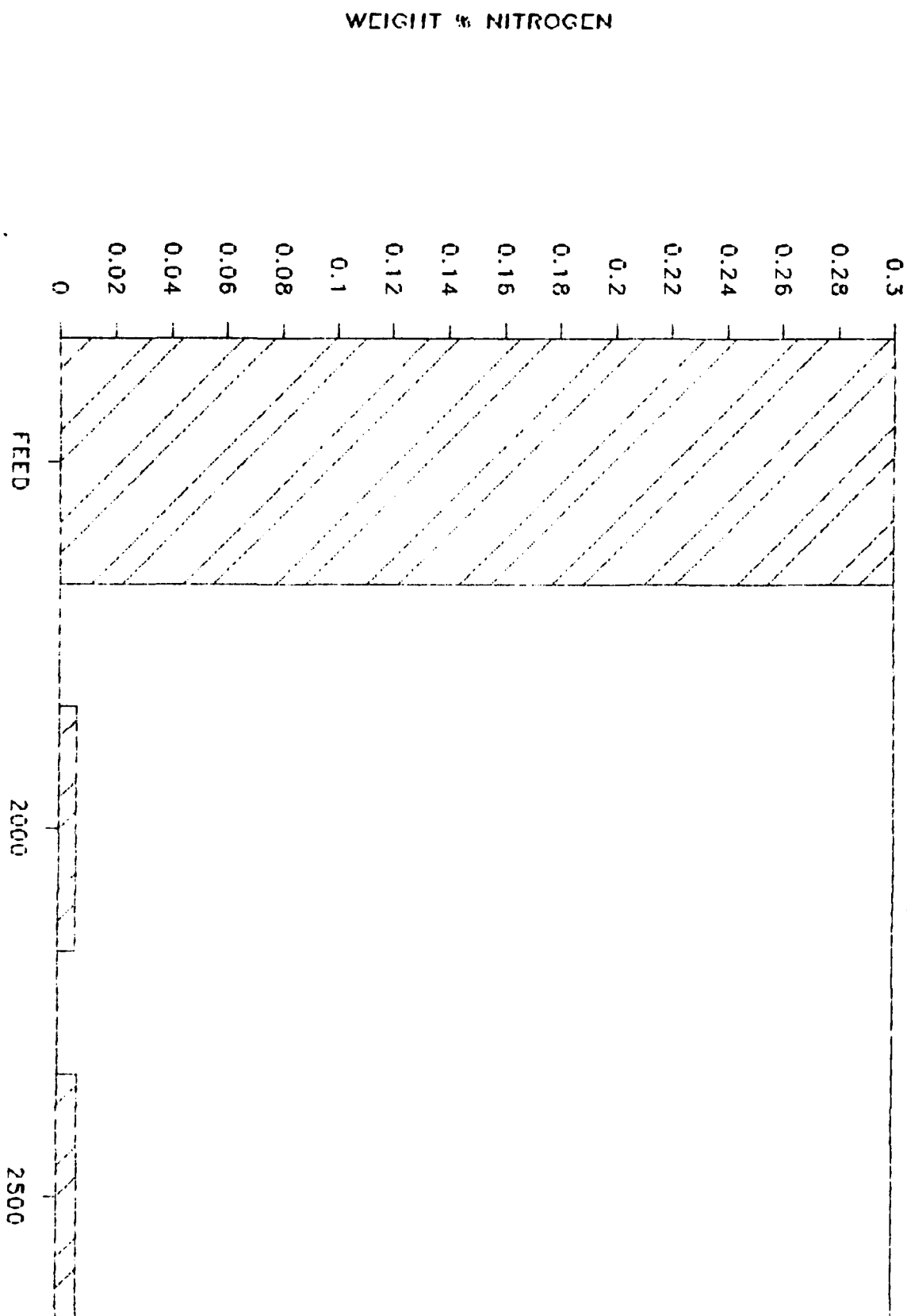
EFFECT OF PRESSURE ON SULFUR

700 F, SHELL CATALYST, LHSV = 1



EFFECT OF PRESSURE ON NITROGEN

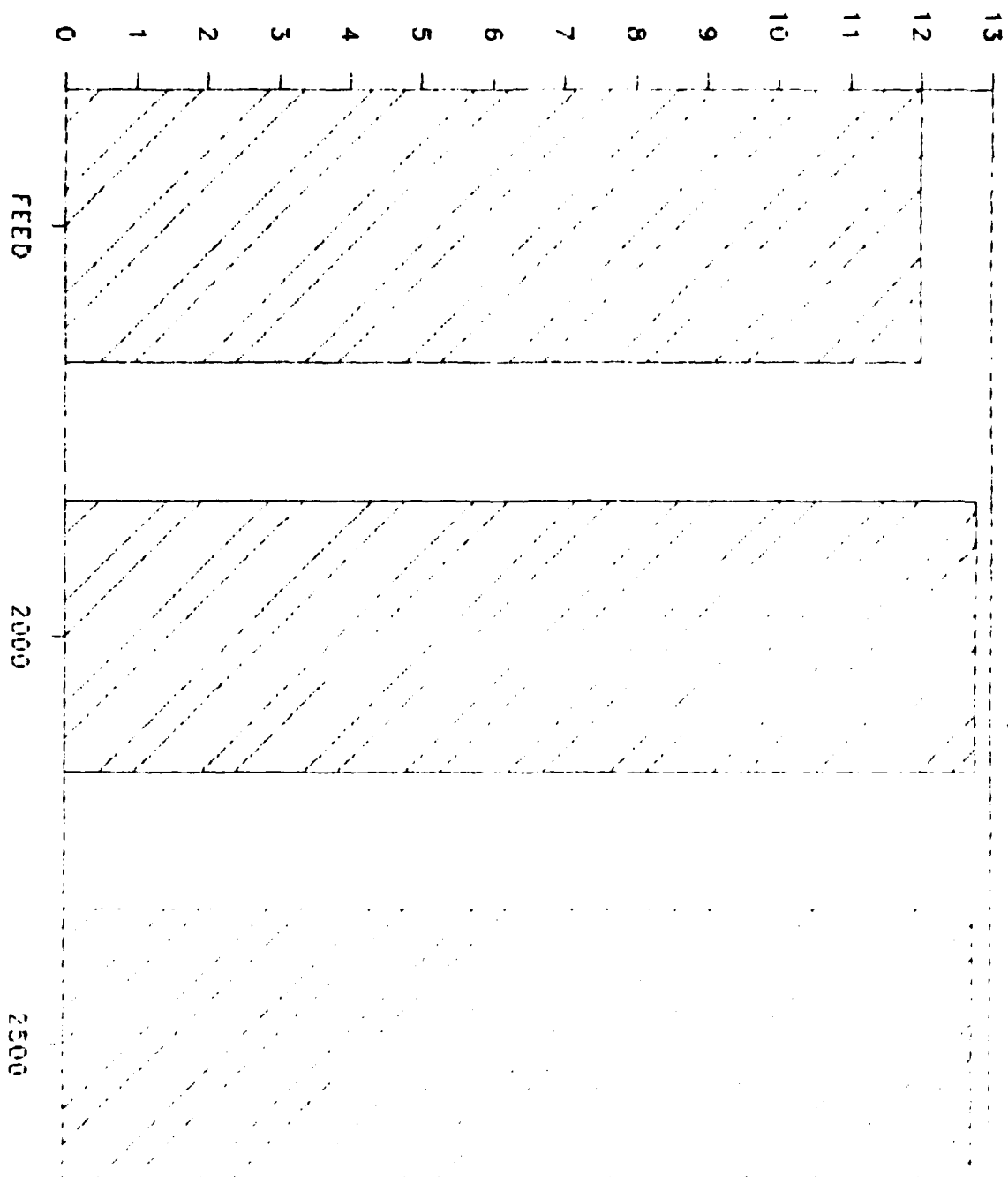
700 F, SHELL CATALYST, LHSV = 1



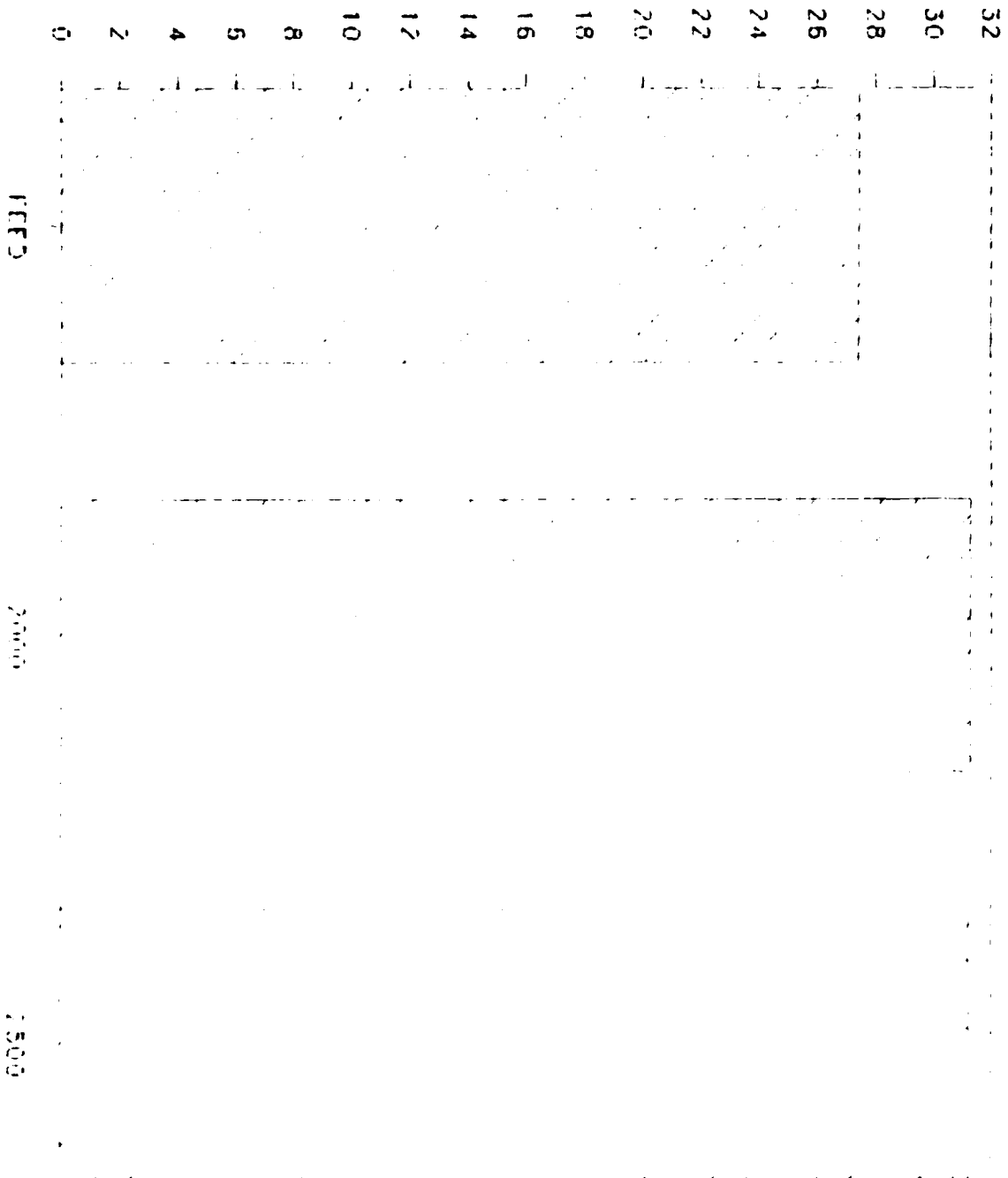
WEIGHT % HYDROGEN

EFFECT OF PRESSURE ON η_{sp}/c

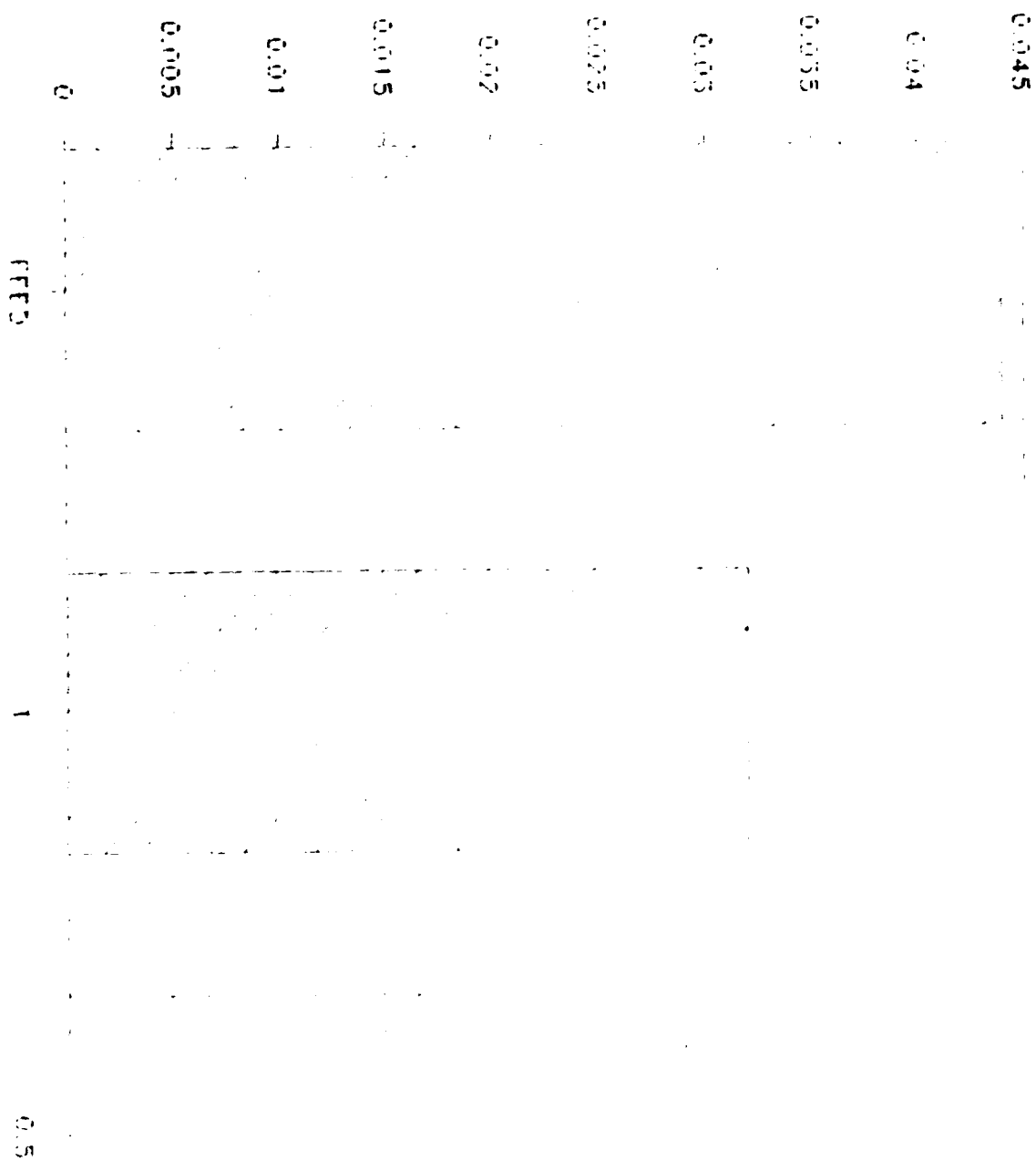
700 F. SHELL CATALYST, LHSV = 1



API GRAVITY

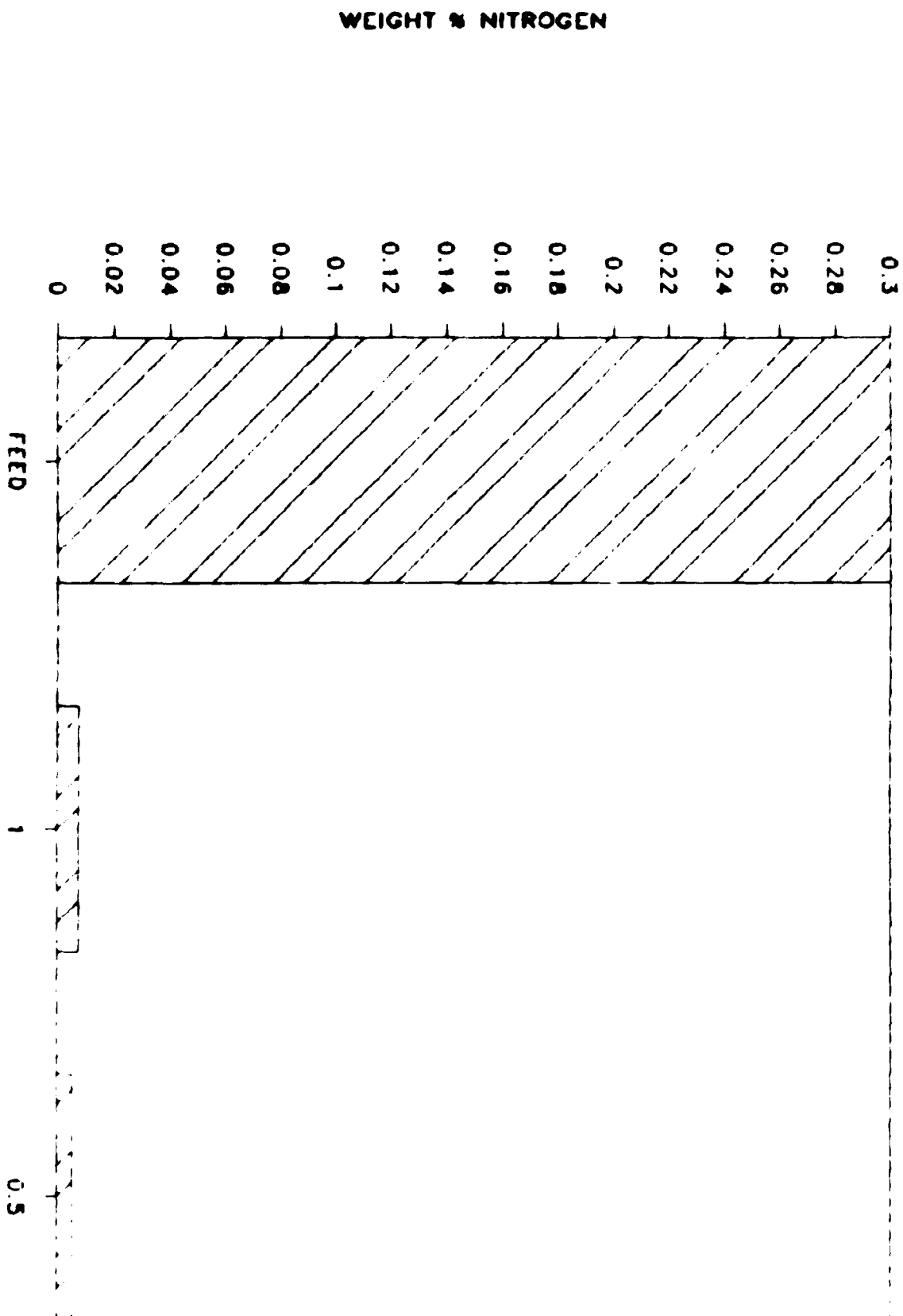


WEIGHT % SULFUR

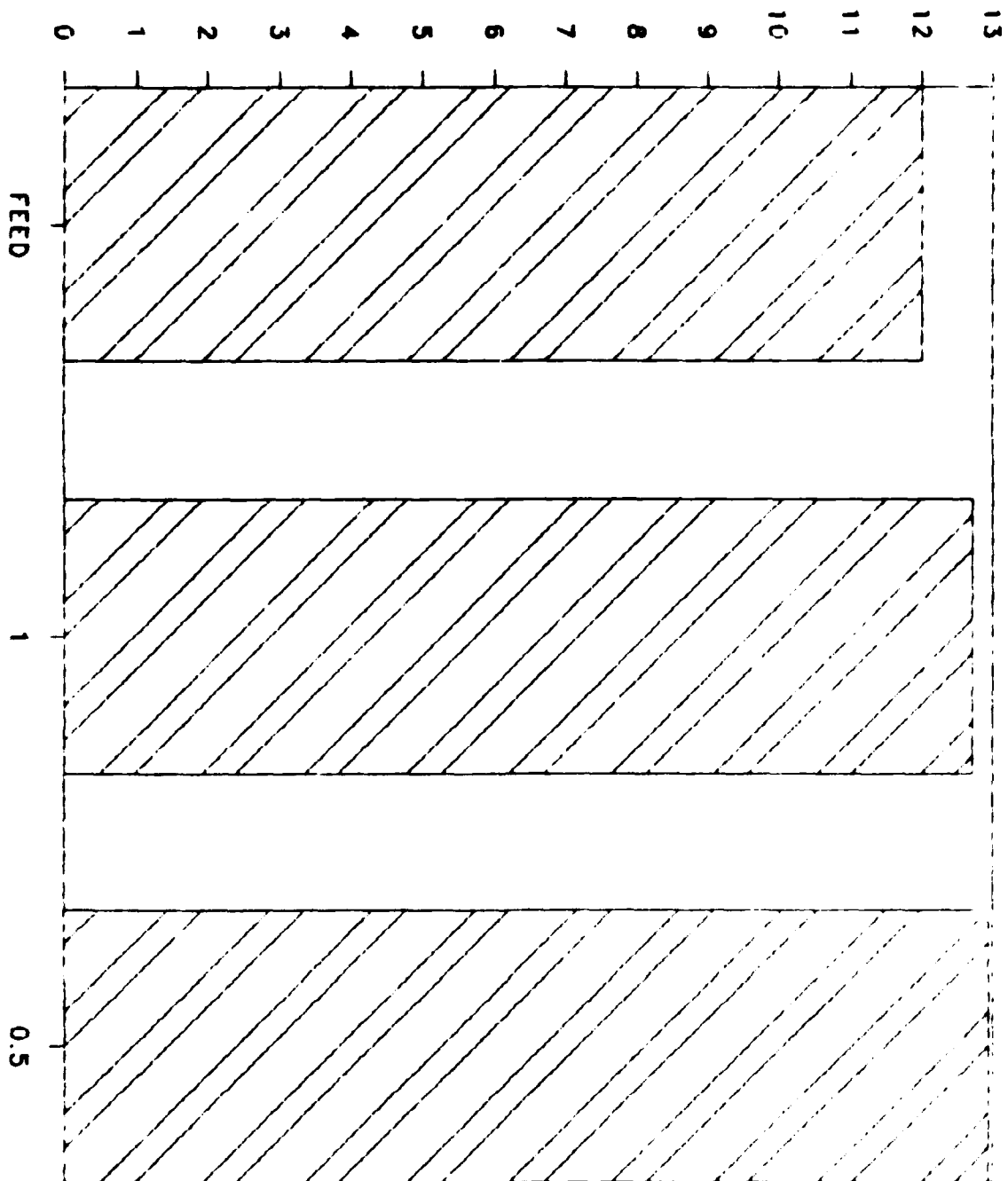


EFFECT OF PRESSURE ON NITROGEN

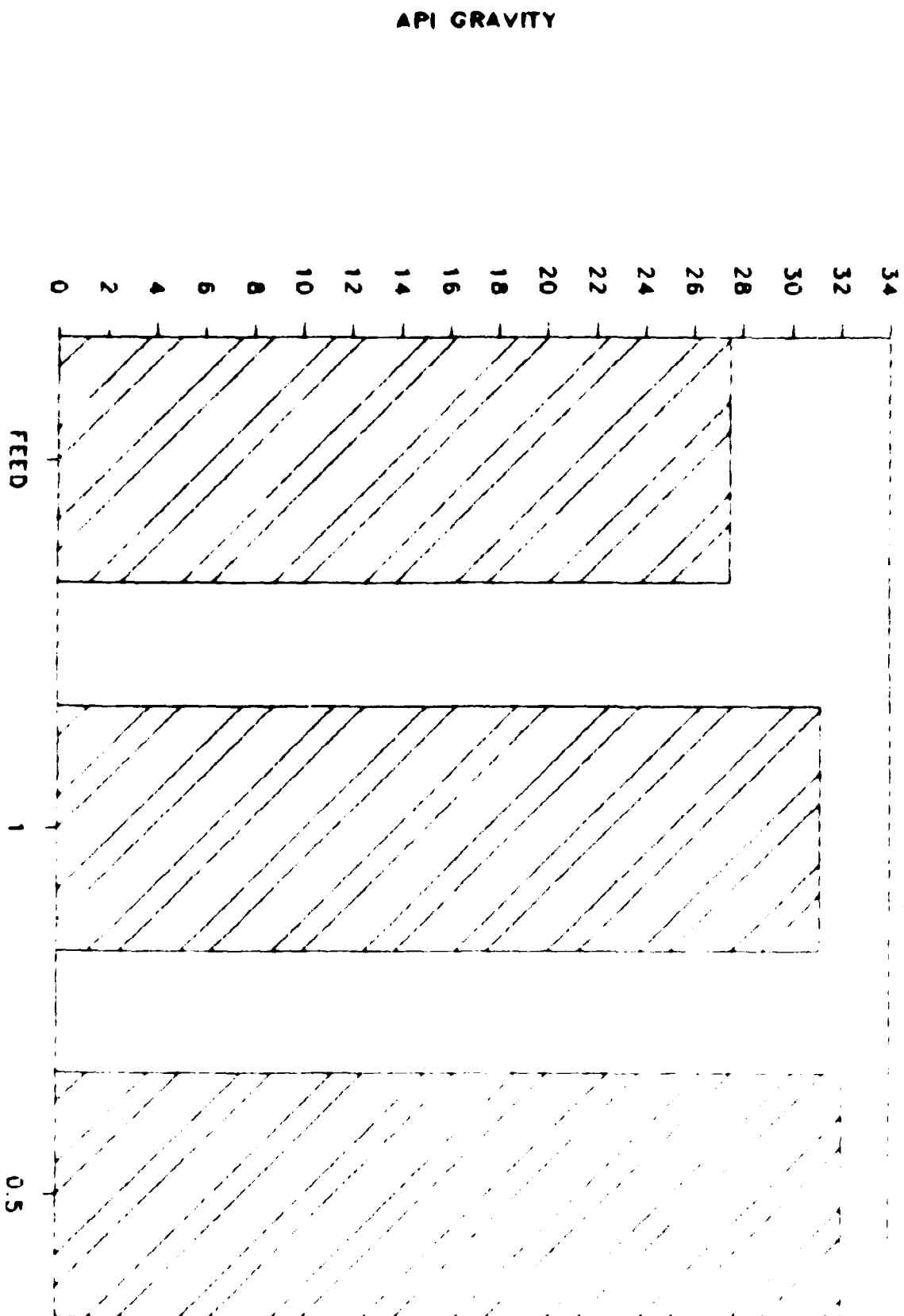
700 F, SHELL CATALYST, 2500 PSIG



WEIGHT % HYDROGEN

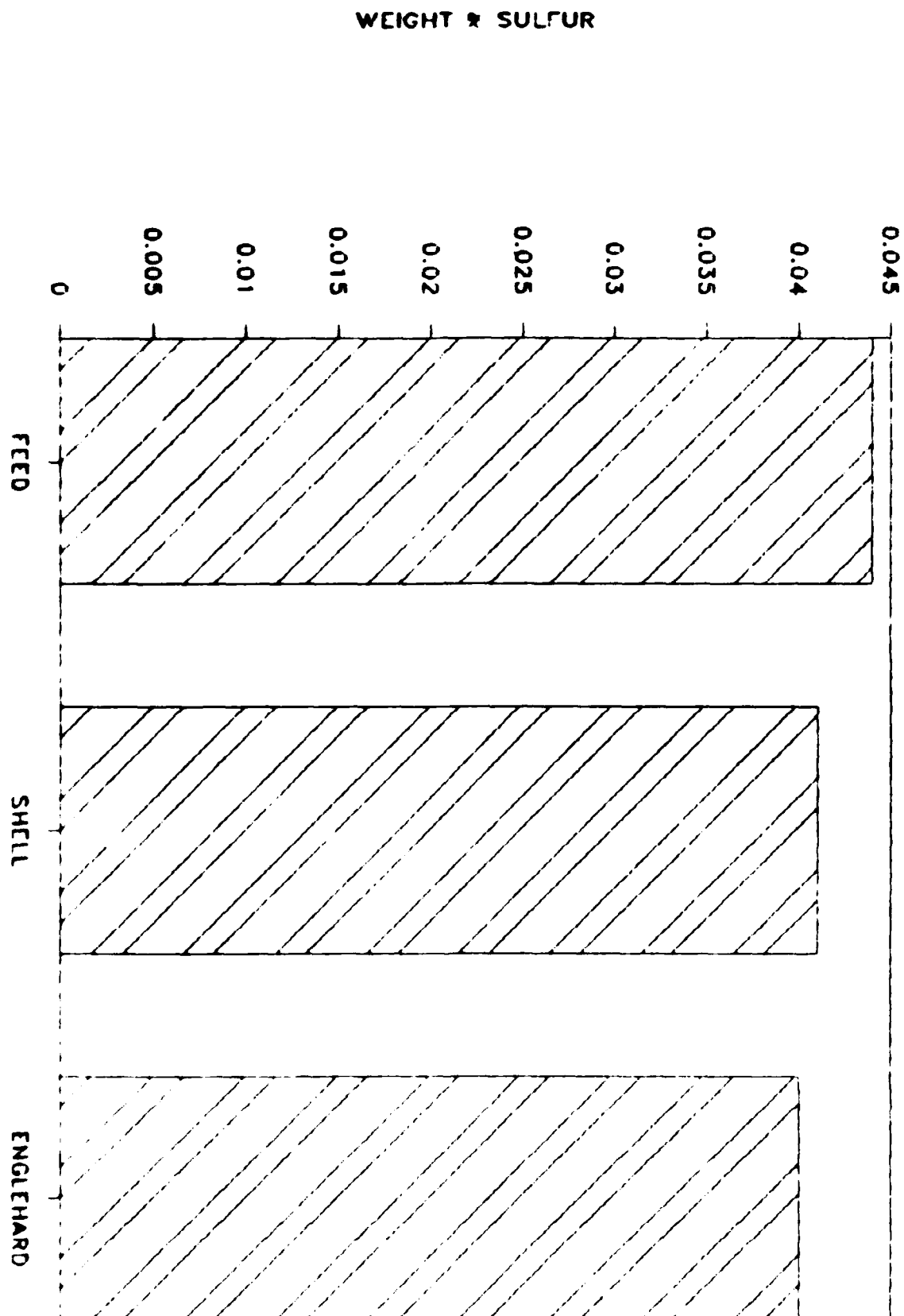


EFFECT OF PHASE SEPARATION ON API GRAVITY 700 F. SHELL CATALYST, 2500 PSIG



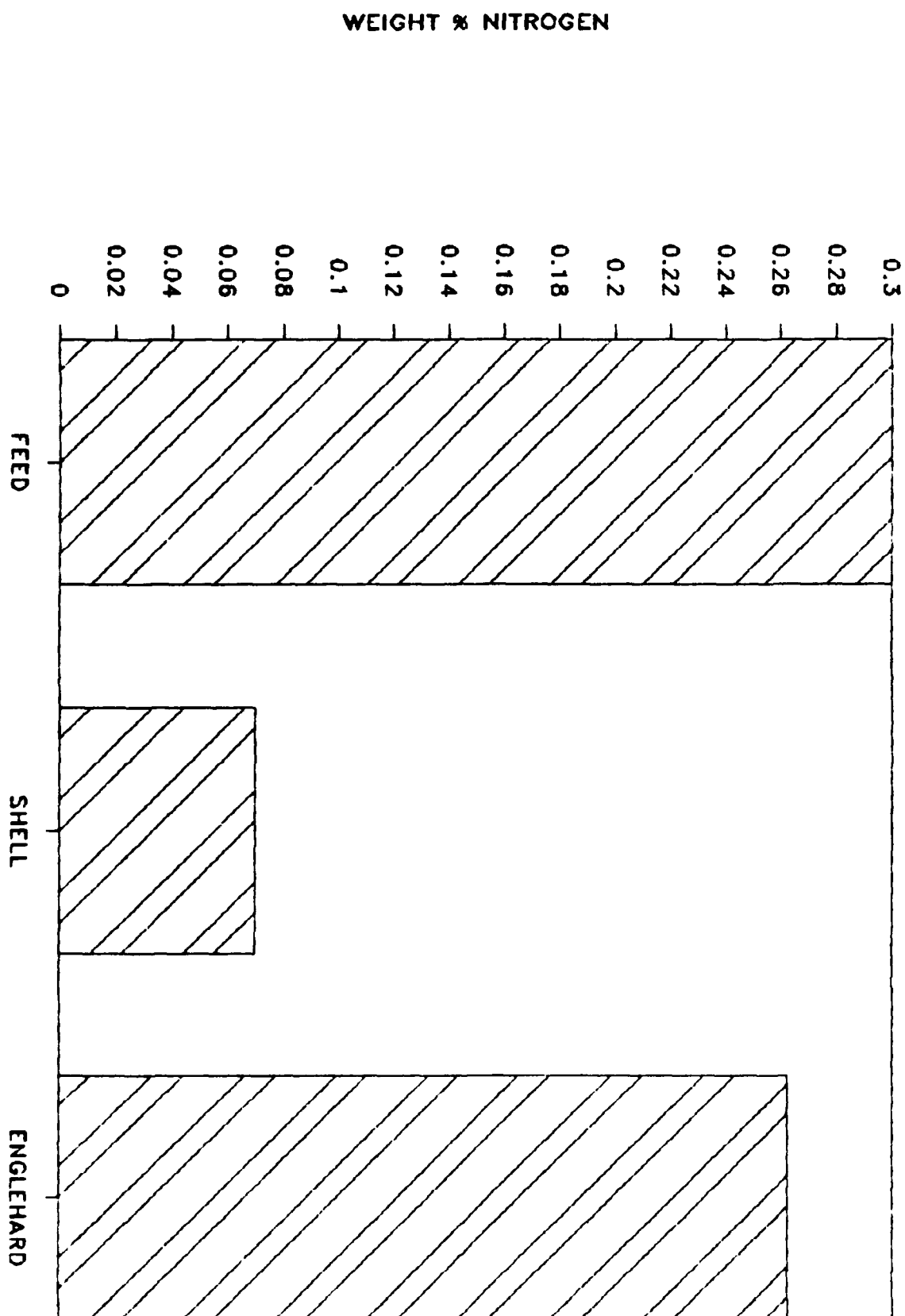
EFFECT OF CATALYST TYPE ON SULFUR

2000 PSIG, 600 F, LHSV = 1



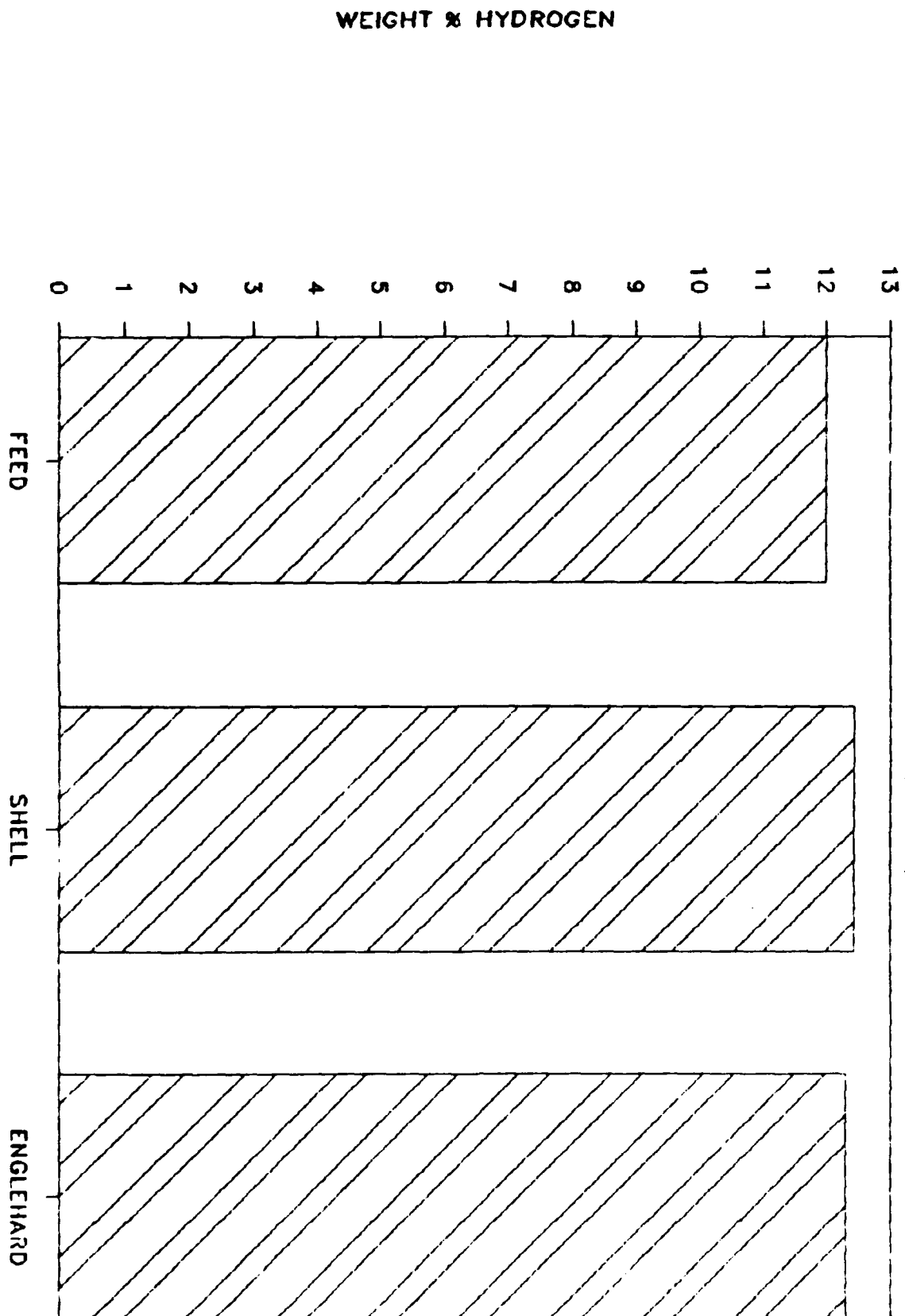
EFFECT OF CATALYST TYPE ON NITROGEN

2000 PSIG, 600 F, LHSV = 1



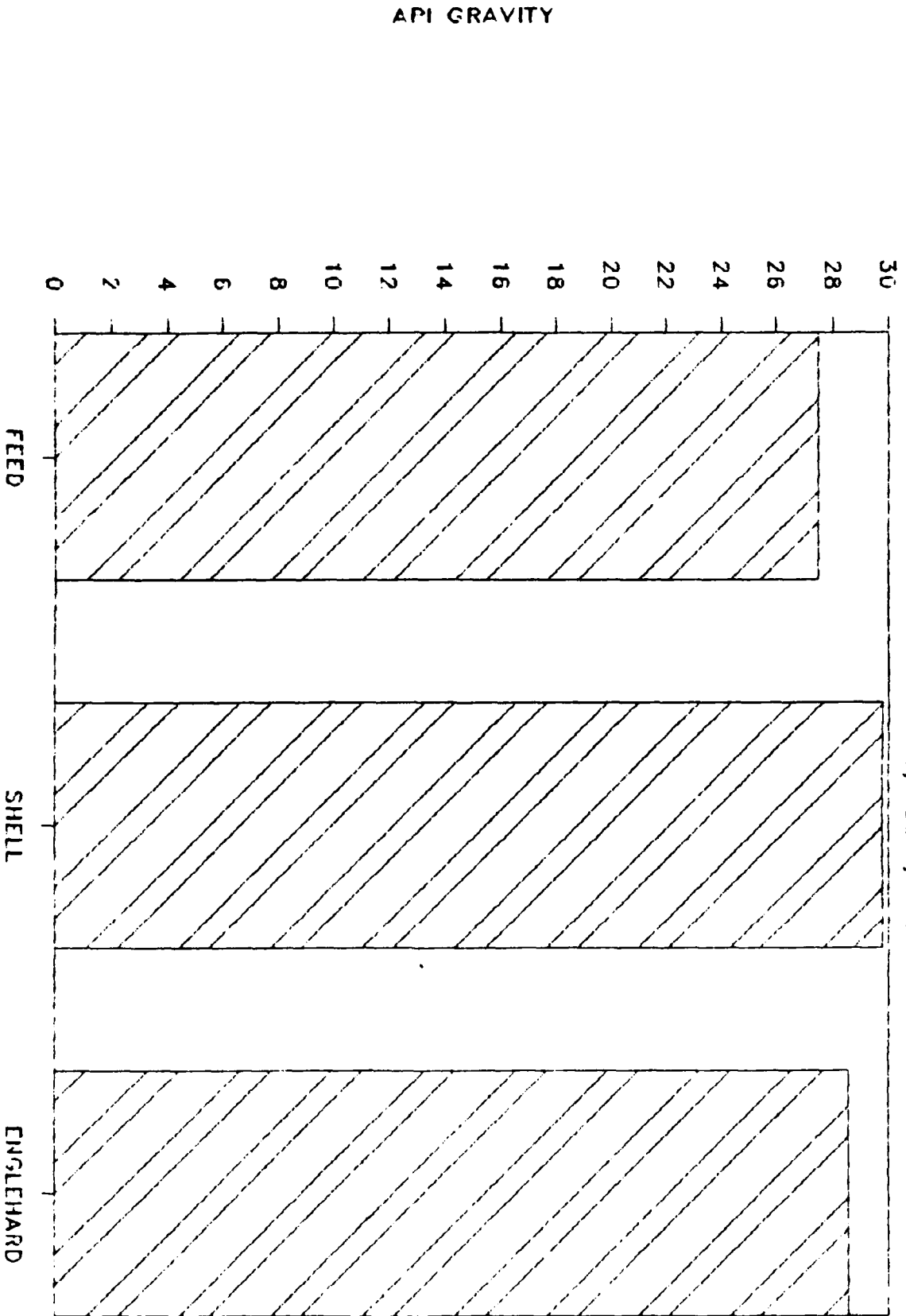
EFFECT OF CATALYST TYPE ON HYDROGEN

2000 PSIG, 600 F, LHSV = 1



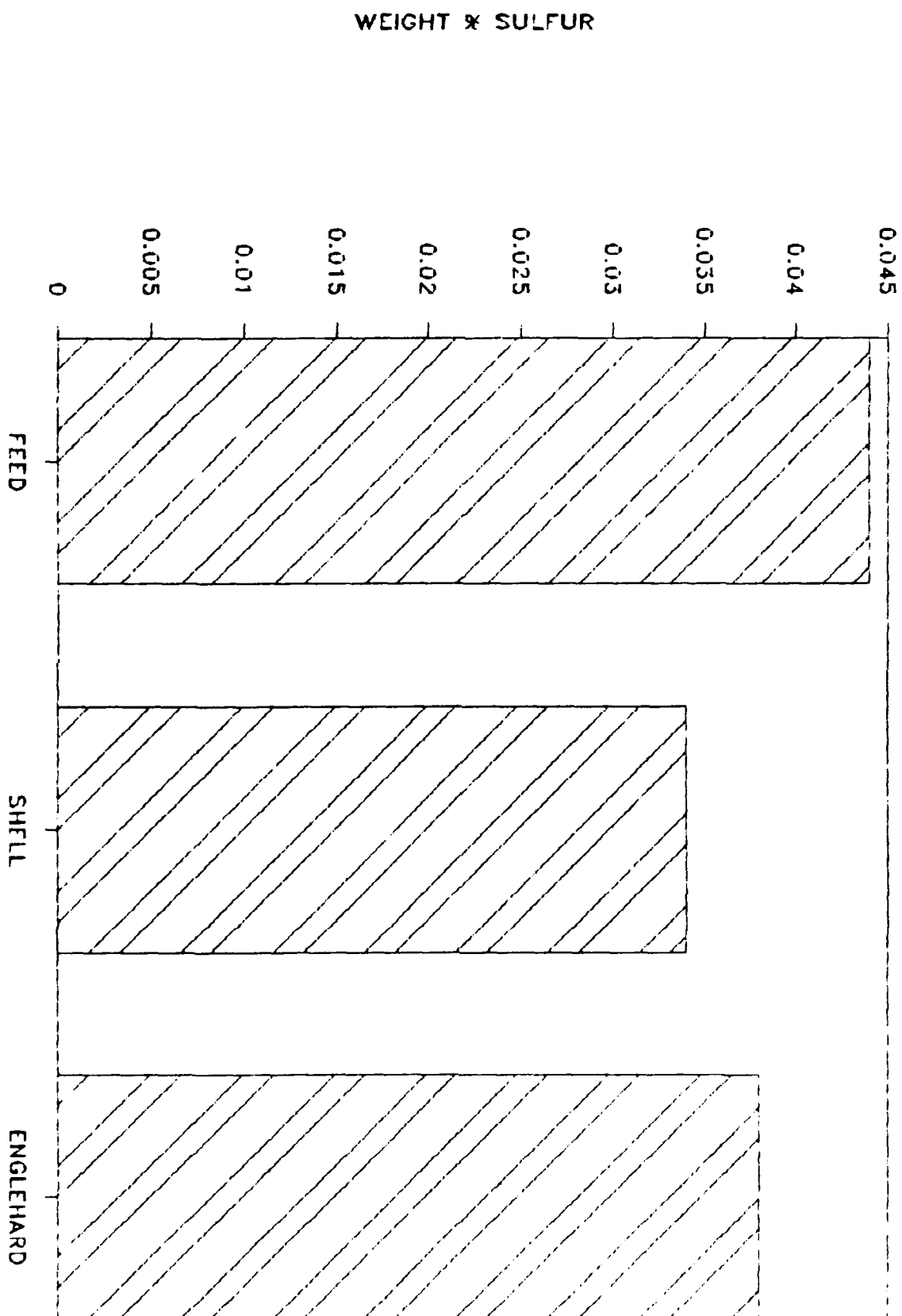
EFFECT OF CATALYST TYPE ON DENSITY

2000 PSIG, 600 F, LHSV = 1



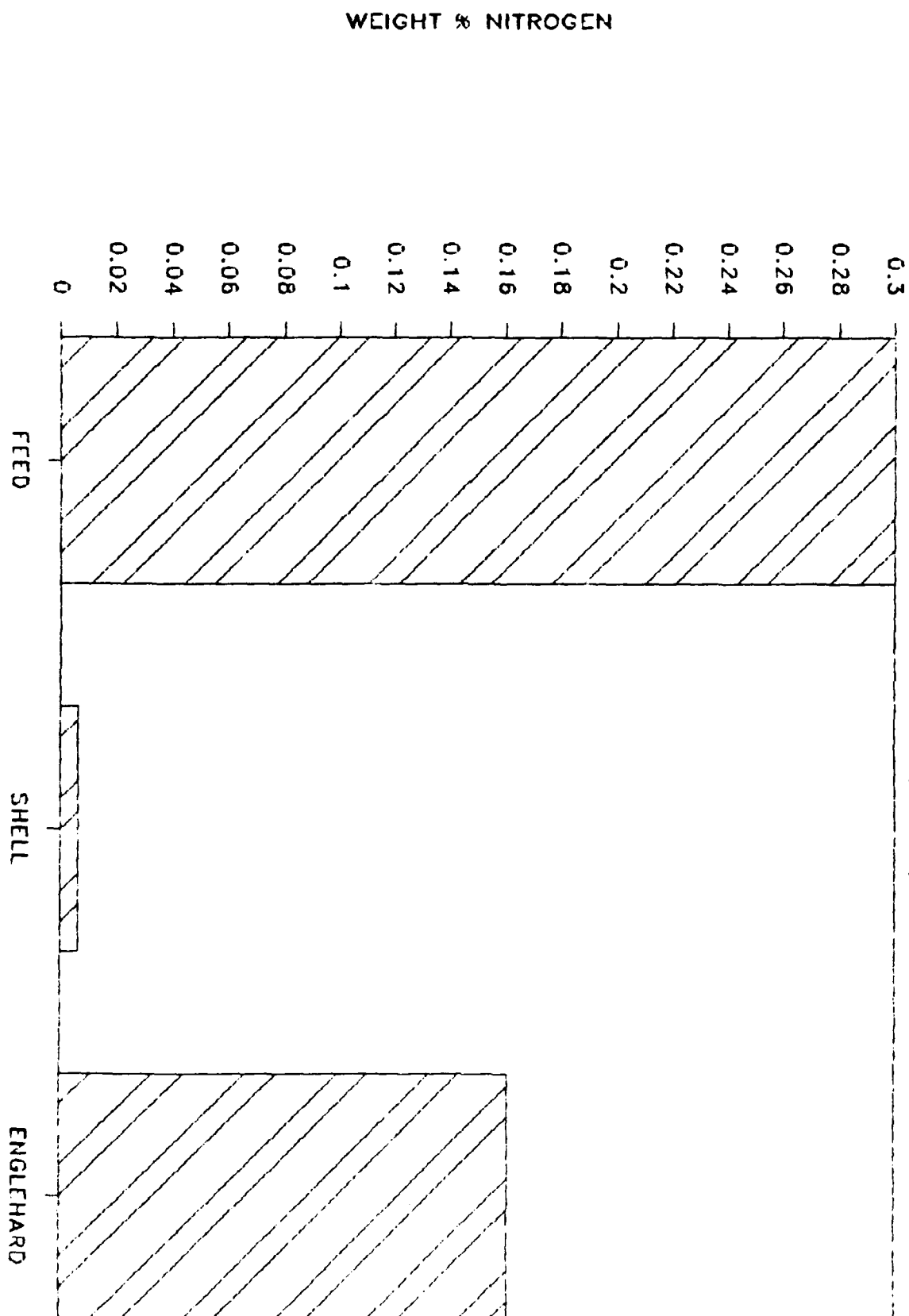
EFFECT OF CATALYST TYPE ON SULFUR

2000 PSIG, 700 F, LHSV = 1



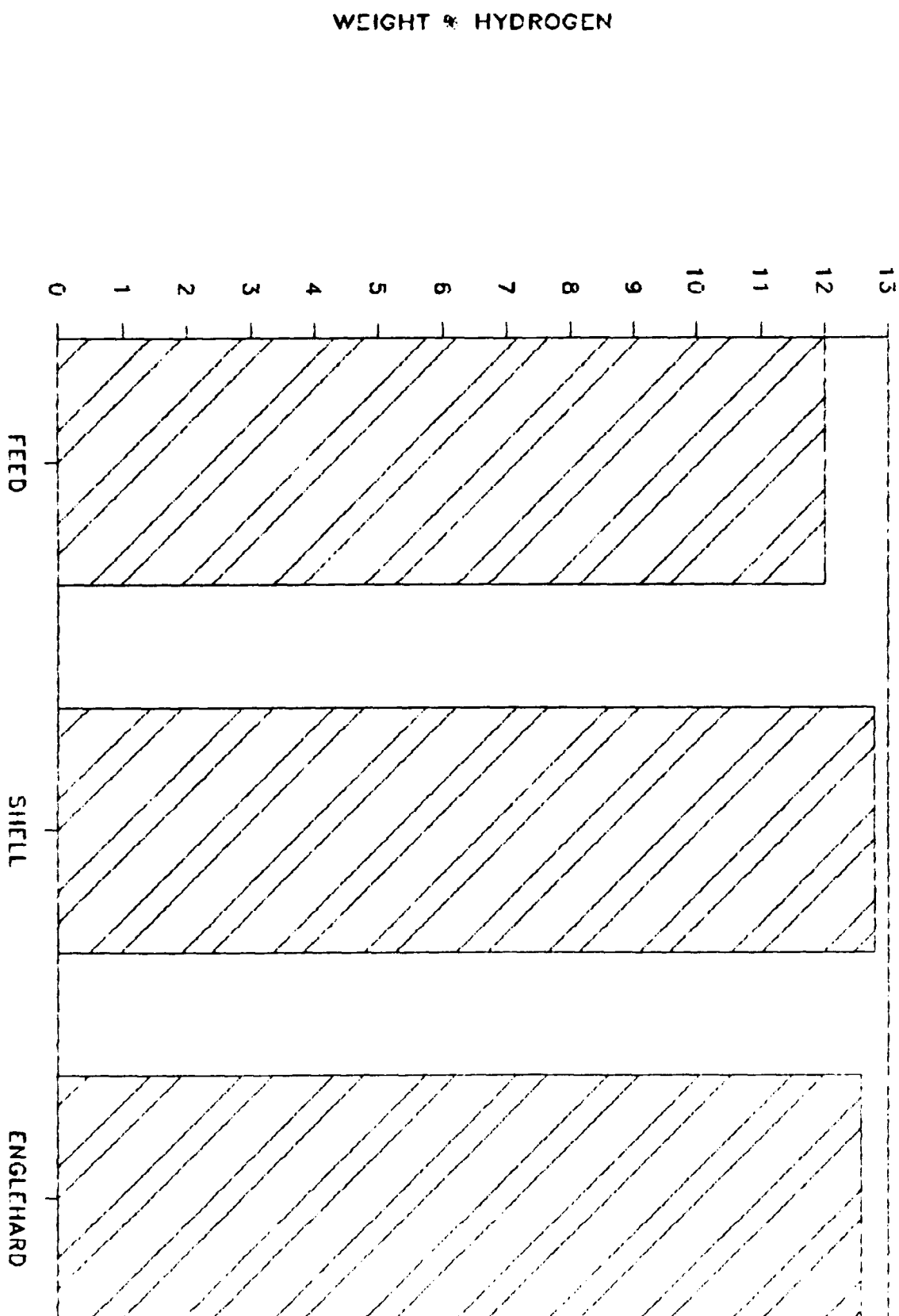
EFFECT OF CATALYST TYPE ON NITROGEN

2000 PSIG, 700 F, LHSV = 1



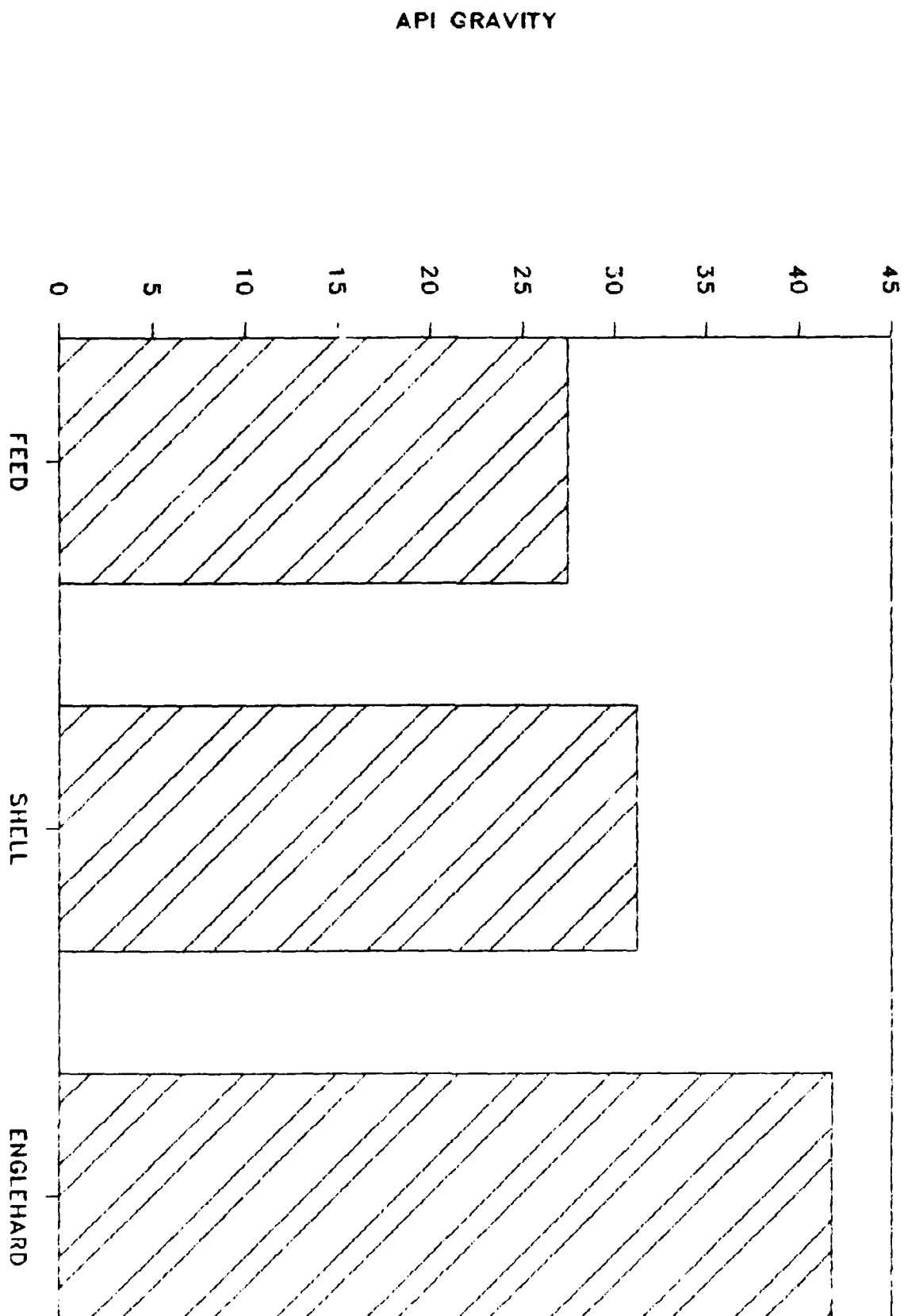
EFFECT OF CATALYST TYPE ON HYDROGEN

2000 PSIG, 700 F, LHSV = 1



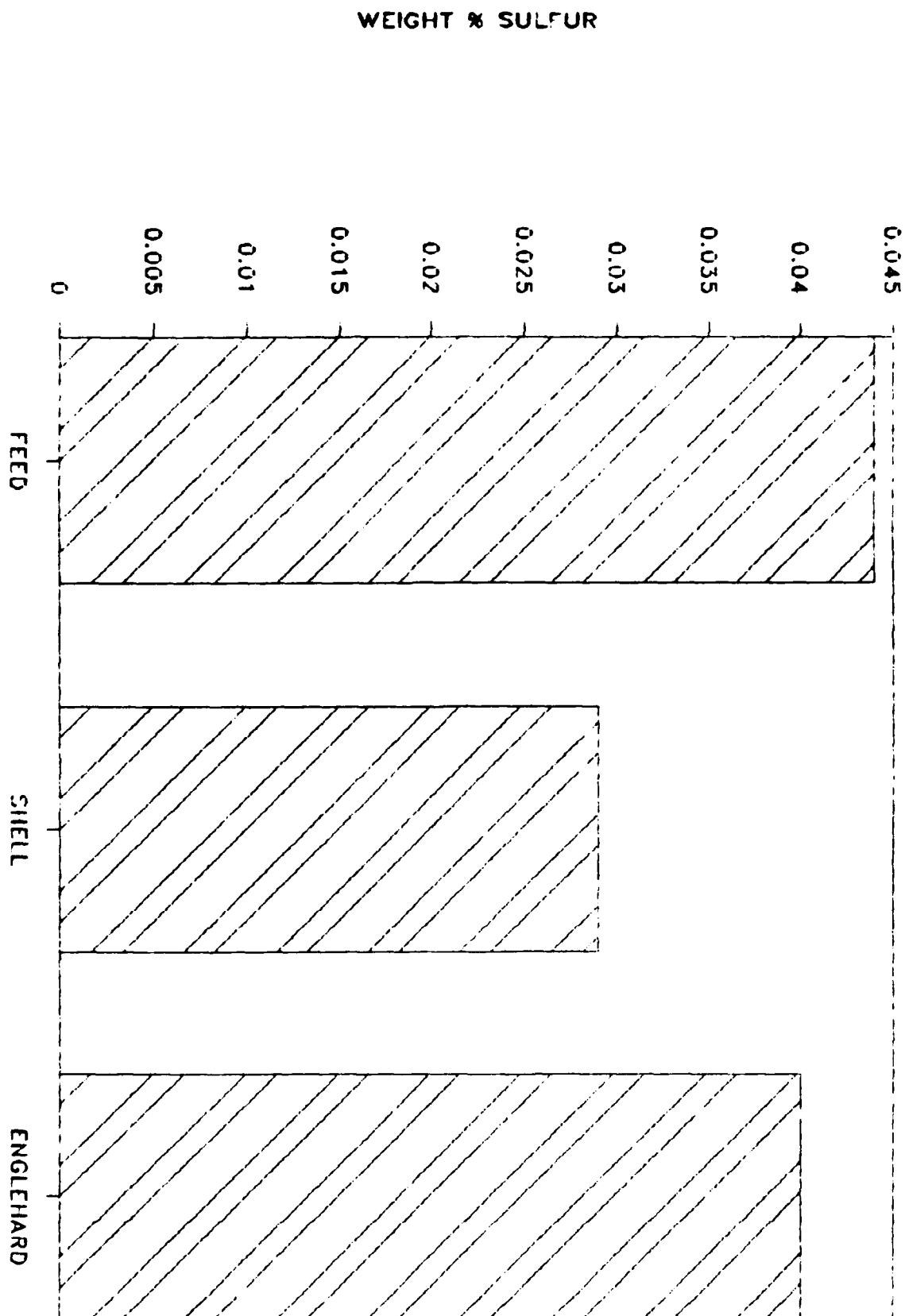
EFFECT OF CATALYST TYPE ON DENSITY

2000 PSIG, 700 F, LHSV = 1



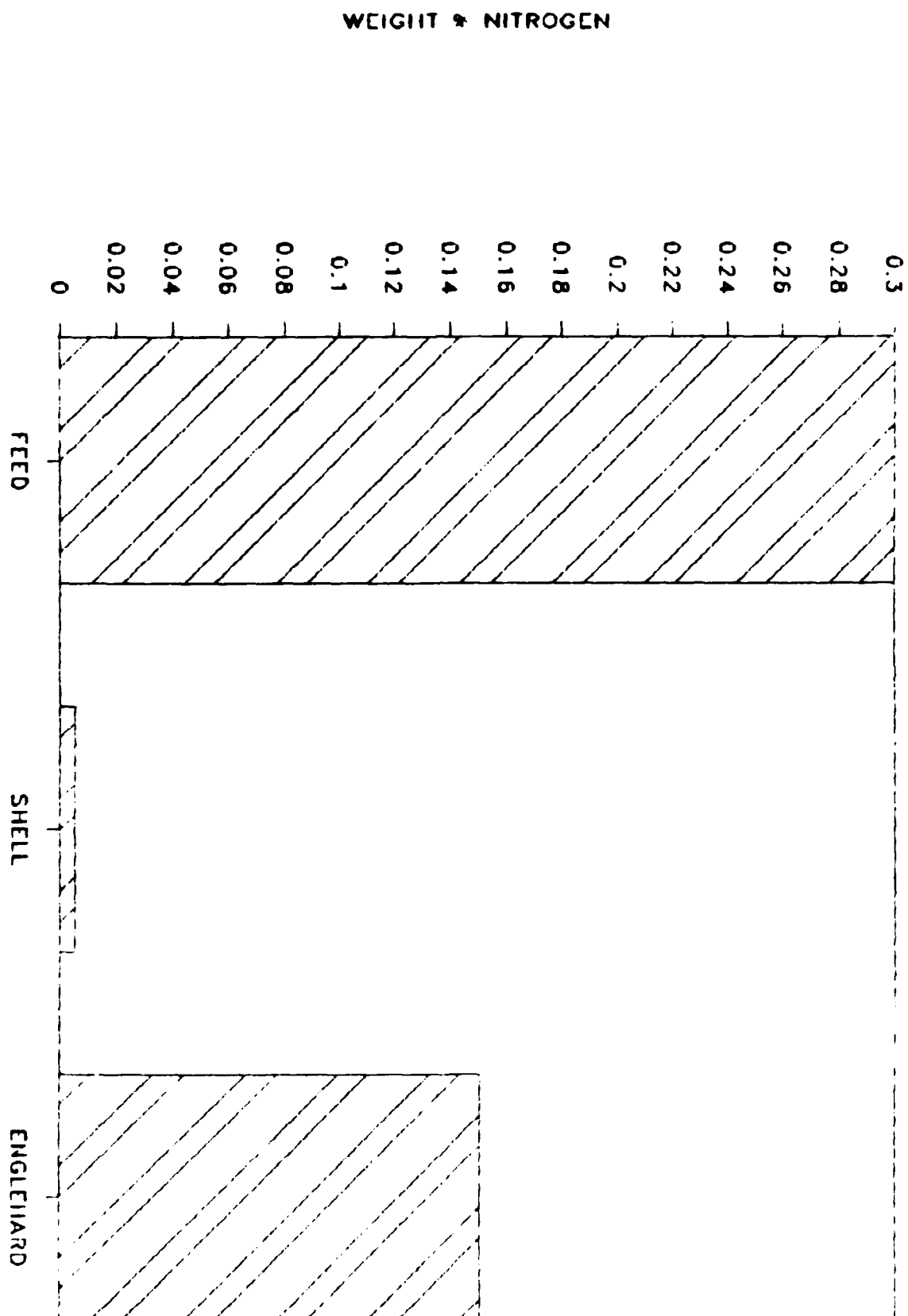
EFFECT OF CATALYST TYPE ON SULFUR

2000 PSIG, 700 F, LHSV = .5



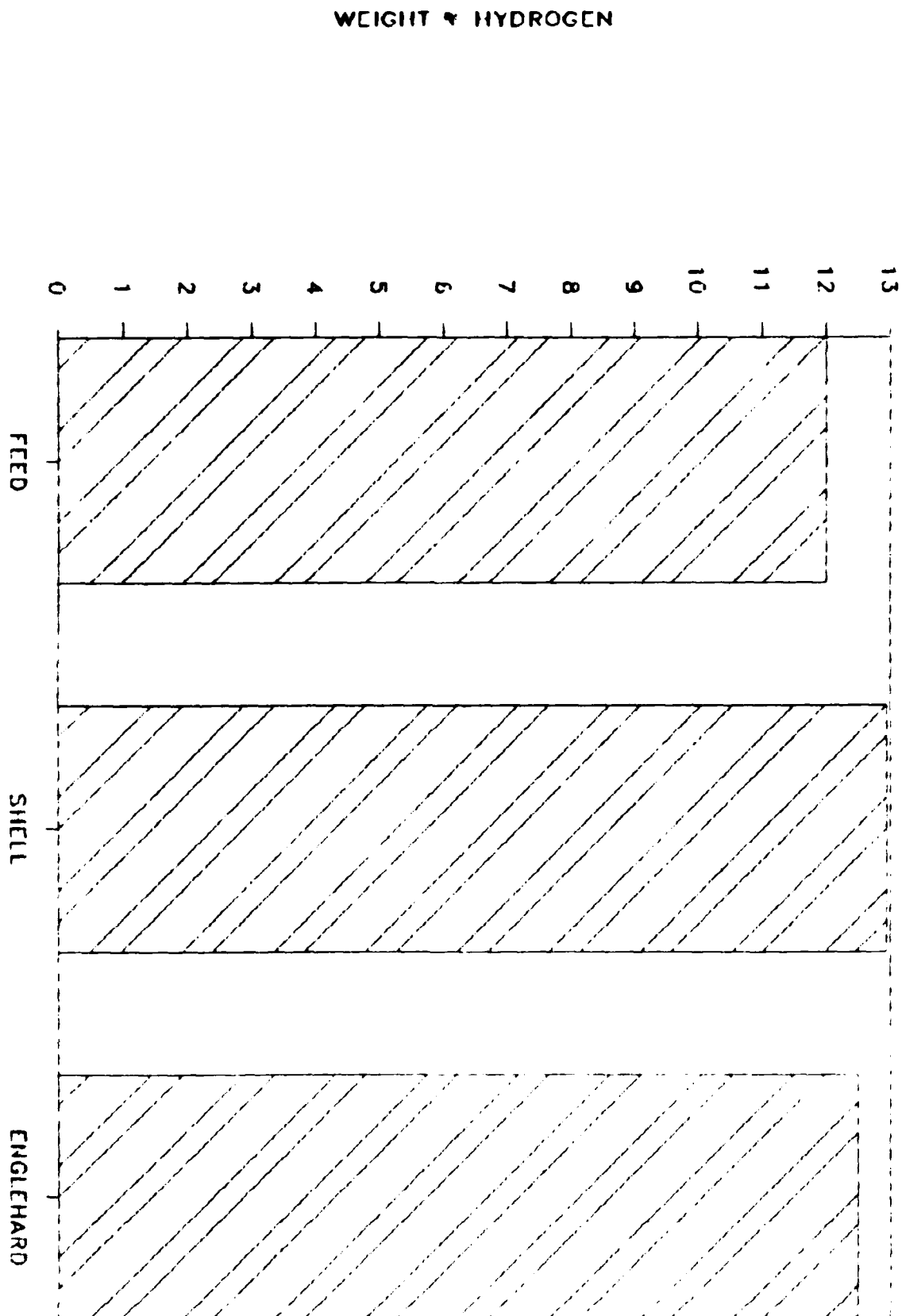
EFFECT OF CATALYST TYPE ON NITROGEN

2000 PSIG, 700 F., LHSV = .5



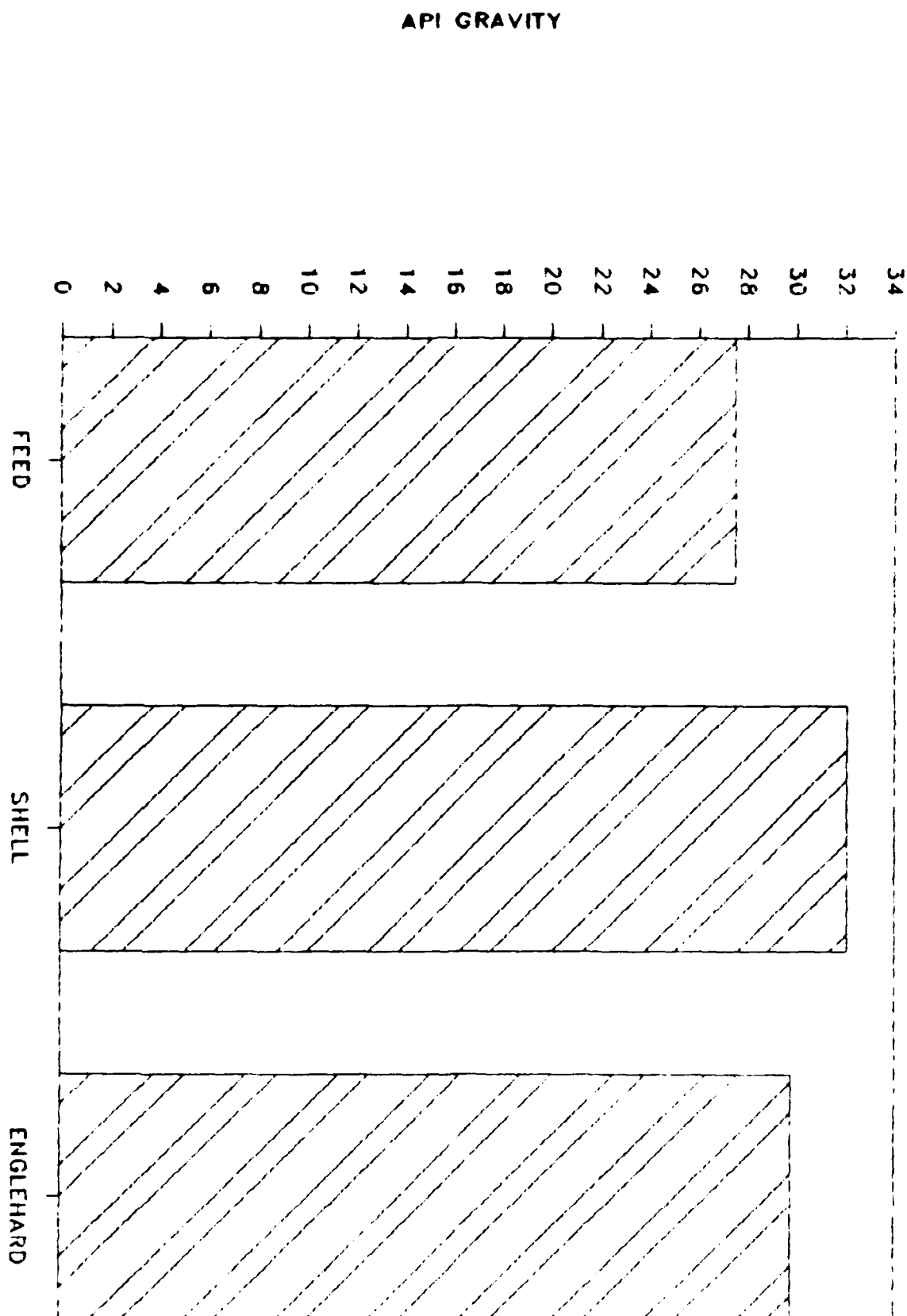
EFFECT OF CATALYST TYPE ON HYDROGEN

2000 PSIG, 700 F, LHSV = .5



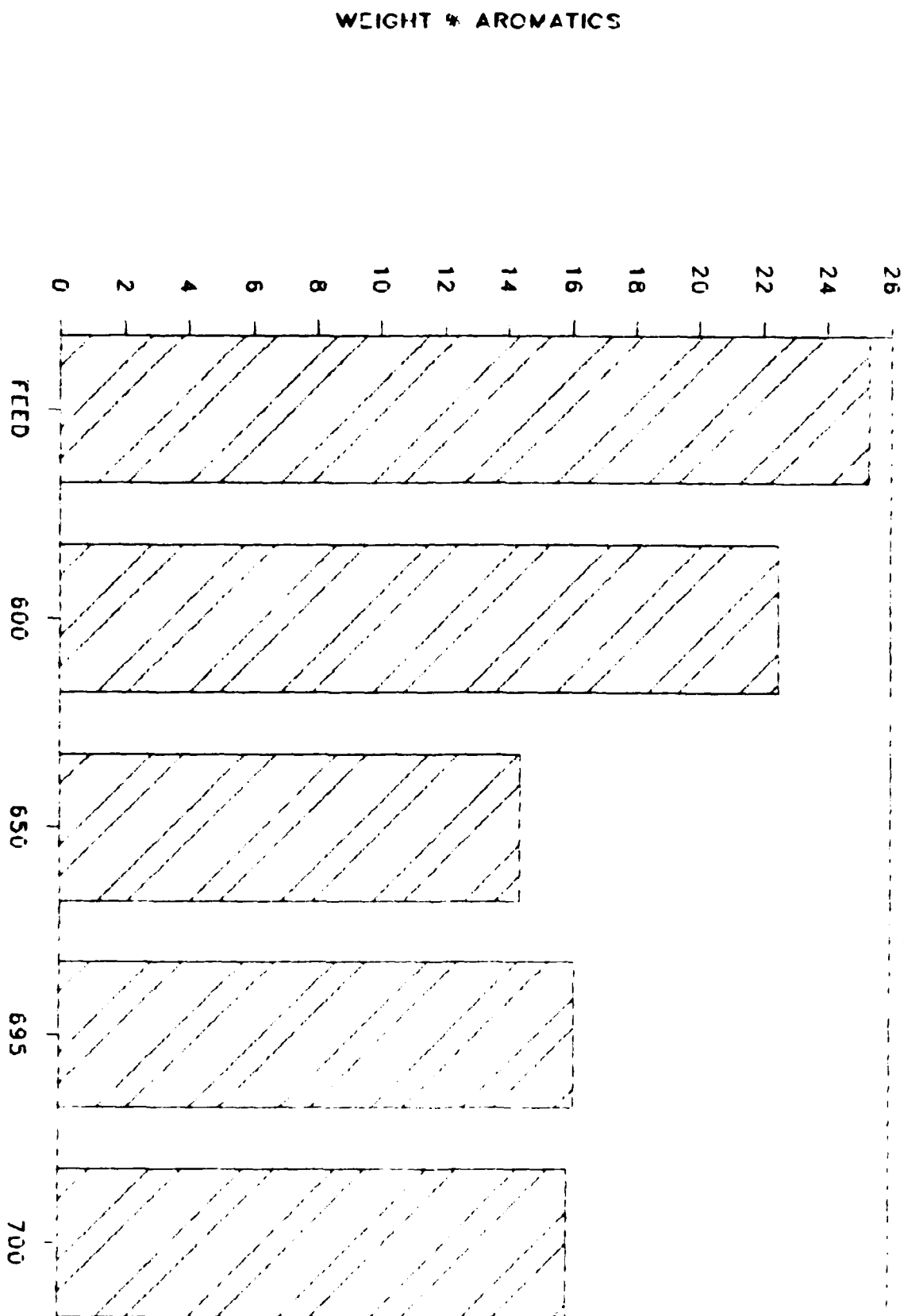
EFFECT OF CATALYST TYPE ON DENSITY

2000 PSIG, 700 F, LHSV = .5



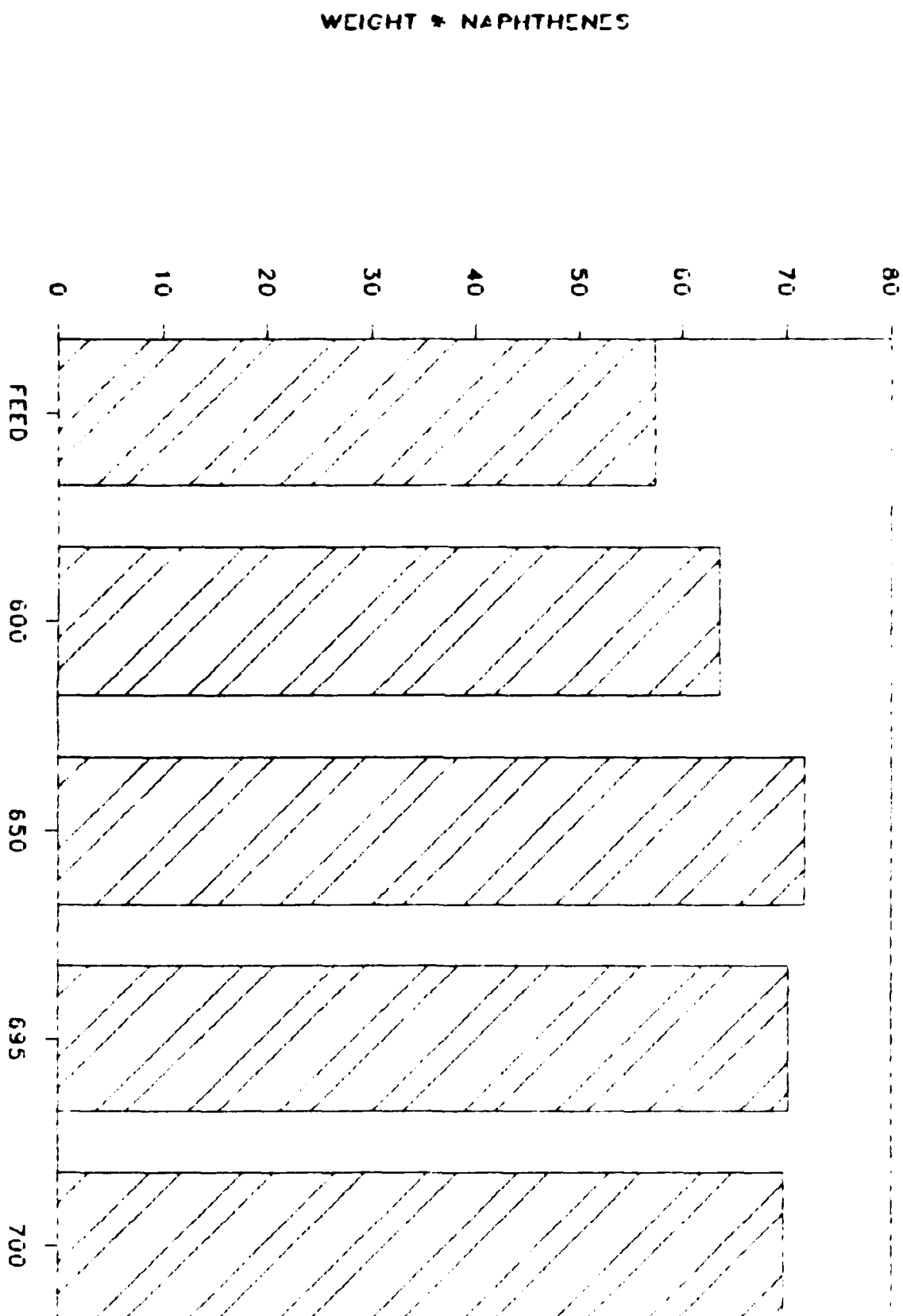
EFFECT OF TEMPERATURE ON AROMATICS

LHSV = 1.0, SHELL CATALYST, 2000 PSIG



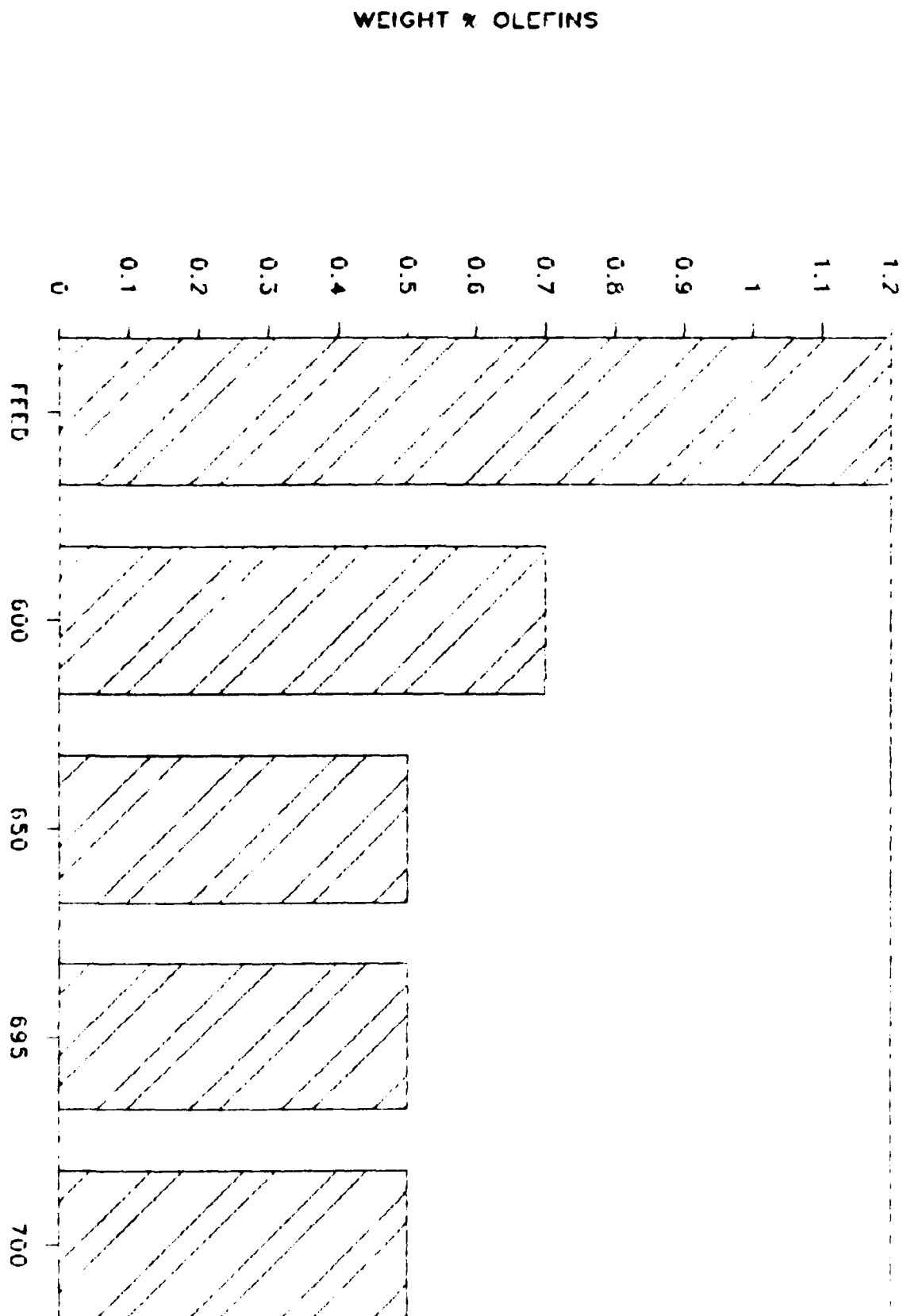
EFFECT OF TEMPERATURE ON NAPHTHENES

LHSV - 1.0, SHELL CATALYST, 2000 PSIG



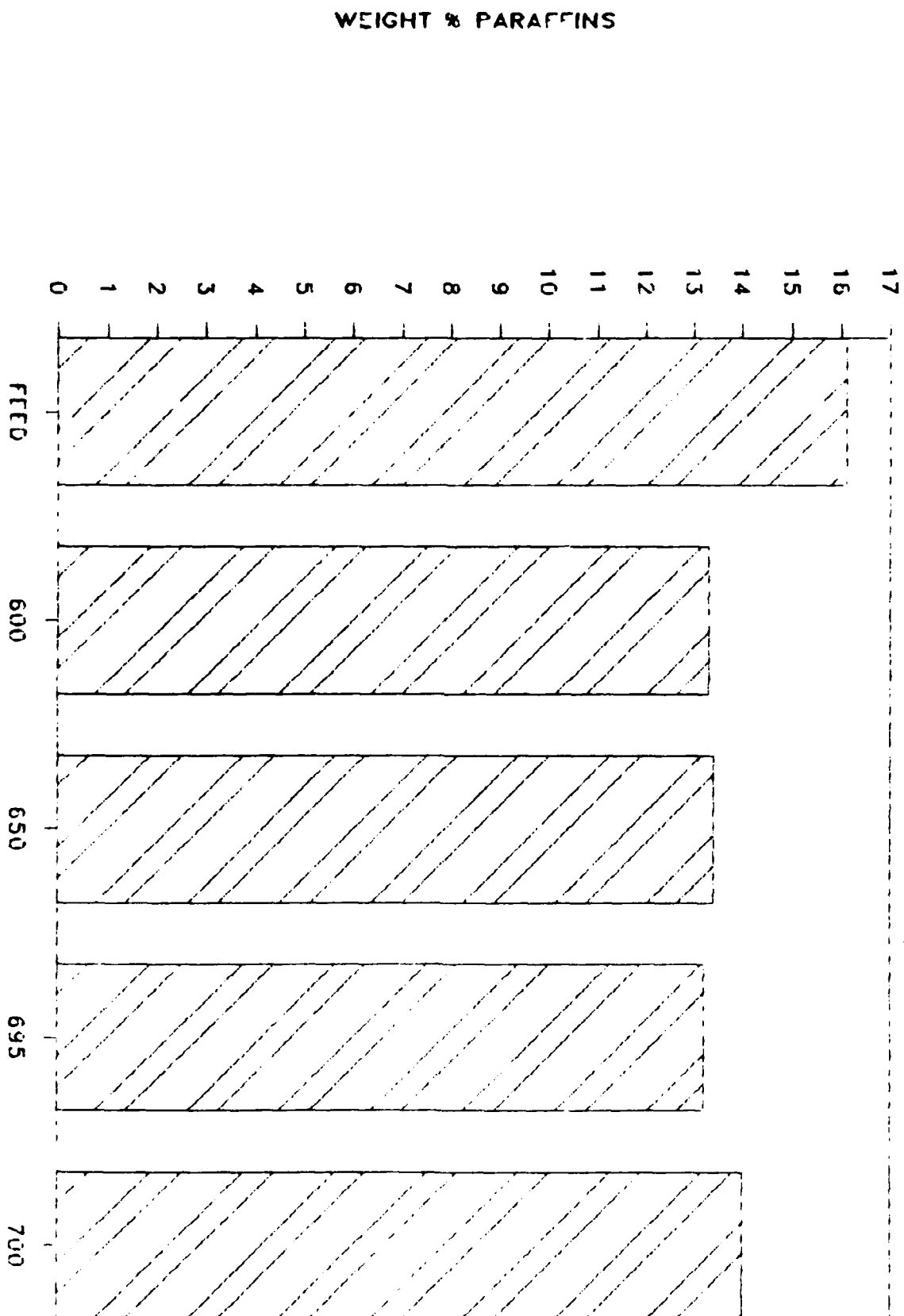
EFFECT OF TEMPERATURE ON OLEFINS

LHSV = 1.0, SHELL CATALYST, 2000 PSIG



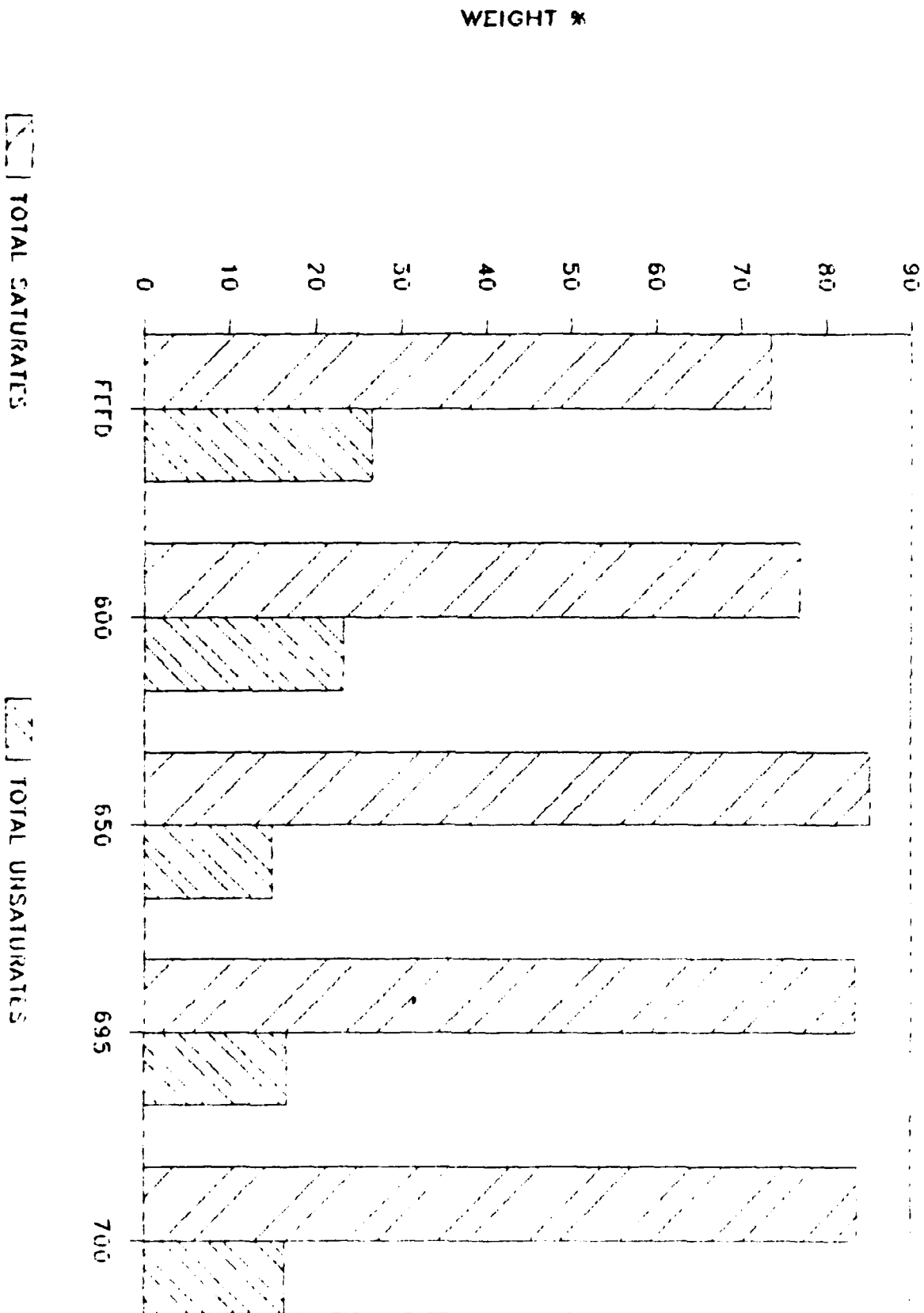
EFFECT OF TEMPERATURE ON PARAFFINS

LHSV = 1.0, SHELL CATALYST, 2000 PSIG



TEMP VS. SATURATES AND UNSATURATES

LHSV = 1.0, SHELL CATALYST, 2000 PSIG



A P P E N D I X B



CATALYTIC, INC., P. O. Box 239, Wilsonville, AL (205) 669-6747

3 October 1986

Carl Smits
J & A Associates
18200 West Highway 72
Golden, CO 80401

Dear Mr. Smits:

Attached is analysis of subbituminous solvent shipped to you on
23 September 1986.


W. R. Hollenack
Assistant Plant Manager

WRH:mp

cc: J. R. Gough
T. W. Johnson
H. D. Schindler
J. Downer

Note: Subbituminous coal feedstock was from Wyodak Upper
Seam, produced from Kerr-McGee mine near Gillete, WY.

The liquid was produced on Run #251 of the Advanced
Coal Liquefaction Facility.

Elemental Analysis:

wt %
 C 87.19
 H 12.40
 N 0.23
 S 0.01
 H₂O 0.05
 Sp. Gravity 0.8862

BTU Content = 20394.29 BTU/lb
 (Calculated using Elemental Analysis Number)

GC SIMULATED DISTILLATION

V160

SN65975

INTEGRATOR II

SAMPLE DATE : 9-18-86

GC RUN DATE : 9-18-86

SAMPLE TIME : 1615

INITIALS : KJ

LIGHT OIL BOILING FRACTIONS

TEMPERATURE	WT %
IBP - 200 F	6.46
200 - 250 F	7.64
250 - 300 F	5.54
300 - 350 F	6.35
350 - 450 F	12.48
450 - EF F	61.52

RESIDUE 0.

IBP = 97.0 F
 EF = 937.4 F

HEAVY OIL BOILING FRACTIONS

TEMPERATURE	WT %
IBP - 350 F	26.00
350 - 450 F	12.48
450 - 500 F	10.54
500 - 550 F	9.98
550 - 650 F	20.02
650 - 850 F	18.94
850 - EF F	2.04

RESIDUE 0.

IBP = 97.0 F
 EF = 937.4 F

COMPONENT	BP F	WT %	CUM %	RT	KF
UNKNOWN	87.9	.22	.22	72	870
PENTANE	97.0	.72	.93	78	865
UNKNOWN	133.4	.55	1.48	102	880
UNKNOWN	144.1	.20	1.68	109	883
HEXANE	156.2	.80	2.48	117	878
UNKNOWN	164.8	.58	3.06	137	891
CYCLOHEXANE	176.0	3.16	6.22	163	735
UNKNOWN	200.0	.24	6.46	184	904
N-HEPTANE	209.1	.51	6.97	192	700
UNKNOWN	223.9	.02	6.99	247	918
UNKNOWN	227.9	.48	7.47	262	920
TOLUENE	231.1	.21	7.69	274	797
UNKNOWN	235.3	.77	8.46	286	925
PYRIDENE	240.8	4.31	12.76	302	917
UNKNOWN	242.6	.84	13.60	306	928
UNKNOWN	248.4	.49	14.10	319	930
UNKNOWN	266.3	2.68	16.78	359	935
P&M-XYLENE	281.1	.67	17.45	392	931
UNKNOWN	283.9	.16	17.61	404	940
O-XYLENE	291.0	1.40	19.02	435	930
UNKNOWN	299.1	.63	19.64	463	945

B2

UNKNOWN	304.8	.09	19.90	483	947
UNKNOWN	308.6	1.68	21.58	496	948
UNKNOWN	319.5	1.03	22.61	534	951
UNKNOWN	333.6	2.78	25.39	583	954
UNKNOWN	341.9	.61	26.00	612	957
INDAN	350.0	1.10	27.10	640	997
UNKNOWN	354.4	1.11	28.21	658	960
PHENOL	359.6	.11	28.32	679	1059
UNKNOWN	360.3	1.13	29.45	681	962
UNKNOWN	365.2	.47	29.92	695	963
UNKNOWN	376.5	1.67	31.59	727	966
UNKNOWN	388.5	.32	31.90	761	969
UNKNOWN	388.8	.29	32.19	762	969
UNKNOWN	396.2	1.16	33.35	783	971
TETRALIN	405.0	1.68	35.03	808	1022
NAPHTHALENE	424.0	.44	35.47	840	1001
UNKNOWN	428.9	1.21	36.68	859	980
UNKNOWN	434.6	1.12	37.79	881	983
UNKNOWN	444.7	.68	38.48	920	989
UNKNOWN	450.9	1.79	40.27	944	993
QUINOLINE	460.0	.31	40.58	979	1116
2-METHYLNAPHTHALENE	465.8	1.74	42.32	984	970
UNKNOWN	473.8	2.01	44.33	1014	1006
UNKNOWN	481.6	.80	45.13	1043	1013
UNKNOWN	485.0	.68	45.81	1056	1015
BIPHENYL	492.0	1.51	47.32	1082	979
DIPHENYL-ETHER	496.3	1.70	49.02	1104	973
UNKNOWN	501.9	.63	49.65	1120	1031
UNKNOWN	511.3	1.63	51.28	1147	1039
UNKNOWN	517.2	2.56	53.84	1164	1044
ACENAPHTHENE	531.1	.75	54.59	1204	1114
UNKNOWN	536.0	1.90	56.49	1217	1060
UNKNOWN	541.0	.78	57.27	1230	1064
DIBENZOFURAN	548.6	1.72	58.99	1250	1123
UNKNOWN	560.7	1.83	60.83	1280	1081
FLUORENE	568.4	1.53	62.35	1299	1137
UNKNOWN	577.3	3.52	65.87	1325	1098
UNKNOWN	586.3	.88	66.76	1351	1108
UNKNOWN	593.8	.52	67.27	1373	1117
UNKNOWN	598.0	2.06	69.33	1385	1122
UNKNOWN	611.0	1.72	71.05	1423	1139
UNKNOWN	615.2	1.58	72.63	1435	1144
UNKNOWN	620.3	1.54	74.17	1450	1151
UNKNOWN	633.4	2.52	76.69	1488	1169
PHENANTHRENE	643.0	1.67	78.36	1516	1187
UNKNOWN	647.0	.65	79.02	1529	1189
UNKNOWN	650.1	1.70	80.72	1539	1194
UNKNOWN	661.5	1.00	81.72	1576	1212
UNKNOWN	665.8	.60	82.31	1590	1220
UNKNOWN	670.8	1.60	83.91	1606	1228
1-METHYLPHENANTHRENE	676.0	1.25	85.16	1623	1358
UNKNOWN	680.7	.75	85.91	1640	1246
9-METHYLANTHRACENE	685.4	.46	86.38	1657	1236
UNKNOWN	689.8	.63	87.01	1669	1262
UNKNOWN	697.9	2.18	89.18	1691	1274
UNKNOWN	711.6	1.17	90.35	1728	1295
FLUORANTHENE	721.9	.79	91.15	1756	1295
UNKNOWN	727.0	1.09	92.24	1771	1320
UNKNOWN	732.5	.49	92.73	1787	1329
PYRENE	740.0	.91	93.64	1809	1365
UNKNOWN	755.6	1.13	94.77	1850	1366
UNKNOWN	761.3	.30	95.07	1865	1374
UNKNOWN	767.0	.19	95.27	1880	1383
UNKNOWN	776.5	.43	95.69	1905	1398
UNKNOWN	783.7	.84	96.54	1924	1409

PERYLENE	838.0	.71	97.93	2087	1538
UNKNOWN	849.9	.03	97.96	2116	1517
UNKNOWN	857.4	.33	98.29	2147	1532
UNKNOWN	857.6	.28	98.57	2148	1533
UNKNOWN	870.2	.07	98.64	2200	1558
UNKNOWN	879.7	.43	99.07	2239	1576
UNKNOWN	906.1	.37	99.44	2348	1618
PERYLENE	937.4	.24	99.68	2477	1651
UNKNOWN	975.0	.17	99.85	2632	1678
UNKNOWN	1020.3	.09	99.94	2819	1759
UNKNOWN	1074.9	.06	100.00	3044	1855



CATALYTIC, INC., P. O. Box 239, Wilsonville, AL 35186 (205) 669-6747

15 December 1986

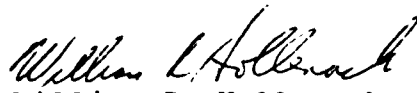
Mr. John Downer
4597 Jupiter Drive
Salt Lake City, Utah 84124

Dear Mr. Downer:

Enclosed are the run conditions and schematic sample location for the sample of solvent sent to you on 23 September 1986. The solvent you received was generated during Run 251-IID period. The solvent was from the low pressure flash (10-15 psig) of the 2nd stage reactor effluent.

If I can be of further assistance please advise.

Sincerely,


William R. Hollenack
Assistant Plant Manager

WRH:mp

cc: J. R. Gough w/out encl.
H. L. Crean w/out encl.
G. A. Styles w/out encl.
T. W. Johnson w/out encl.

Overall TSL Yields
(Phase 3 Data)

Operating Period	251-IIB	251-IIC	251-IID
Date, 1986	8/2-8/5	8/7-8/11	8/14-8/18
Days selected	8/2,8/3 8/4,8/5	8/7,8/8 8/10,8/11	8/14,8/15 8/17,8/18
Coal feed rate, MF lb/hr	353	354	249
Coal conc. in slurry, wt % MF	33.0	33.1	33.3
Process solvent, wt %			
Resid (a)	25(33)	26(34)	26(34)
CI	24	24	24
<u>1st stage</u>			
Reaction temp., °F (average)	819	818	807
Inlet H ₂ part. press., psia	2,510	2,530	2,530
Coal space rate, MF lb/hr/ft ³ (>700°F)	23.7	23.9	16.8
Fe ₂ O ₃ addition, wt % MF coal	0.8	1.5	1.5
<u>2nd stage</u>			
Reaction temp., °F (average)	743	745	745
Inlet H ₂ part. press., psia	2,550	2,560	2,580
Space velocity, hr ⁻¹	2.79	2.79	1.99
Catalyst type	Amocat 1C	Amocat 1C	Amocat 1C
Catalyst age, lb (resid+CI)/lb cat	915-1028	1105-1254	1339-1443
<u>CSD</u>			
DAS type	2204	2204-2254	2254
H ₂ consumption, wt % MAP	6.3±0.1	6.0±0.1	6.3±0.1
Energy rejection, %	12.7±0.7	13.7±1.2	12.6±0.6
<u>Yield, wt % MAP coal</u>			
Water	13.6±1.1	13.0±0.7	14.1±0.5
H ₂ S, CO, CO ₂ , NH ₃	10.1±0.4	10.4±0.8	8.2±0.5
C ₁ -C ₃ gas	8.1±0.3	7.0±0.4	6.0±0.8
C ₄ + distillate	61.0±1.3	58.4±1.6	60.7±1.8
C ₄ + naphtha	19.7±1.1	19.3±1.4	18.9±1.1
Middle distillate	10.3±0.6	11.4±0.7	11.0±0.4
Distillate solvent	31.1±1.9	27.6±0.9	30.8±3.0
Resid (b)	3.9±2.2	7.3±1.8	7.9±1.7
Ash concentrate	9.4±0.8	9.3±1.0	8.7±0.3
<u>H₂ efficiency</u>			
lb C ₄ + dist/lb H ₂ cons	9.7±0.1	9.7±0.3	9.7±0.3
<u>C₁-C₃ selectivity (X100)</u> to C ₄ + distillate	13.0±1.0	12.0±1.0	10.0±2.0
<u>Coal conversion, wt % MAP (c)</u>			
1st stage	94.2±0.8	94.7±1.1	94.4±1.0
1st and 2nd stage	95.4±0.8	95.7±0.4	96.0±0.3
Two stage	95.4±1.3	95.3±0.4	94.7±0.7
<u>(Resid + UC) conversion,</u> <u>wt % feed (d)</u>			
1st stage (e)	34.8±1.1(61.0±1.8)	35.6±1.7(62.3±3.1)	37.0±1.4(64.1±1.8)
2nd stage	23.4±1.3(27.2±1.4)	17.8±2.1(20.1±2.7)	18.4±1.7(20.2±2.3)

(a) Data in parentheses on CI-free basis.

(b) Includes TSL system UC accumulation.

(c) Cresol solubles.

(d) Data in parentheses are based on MAP coal.

(e) MAP coal as 100 wt % UC.

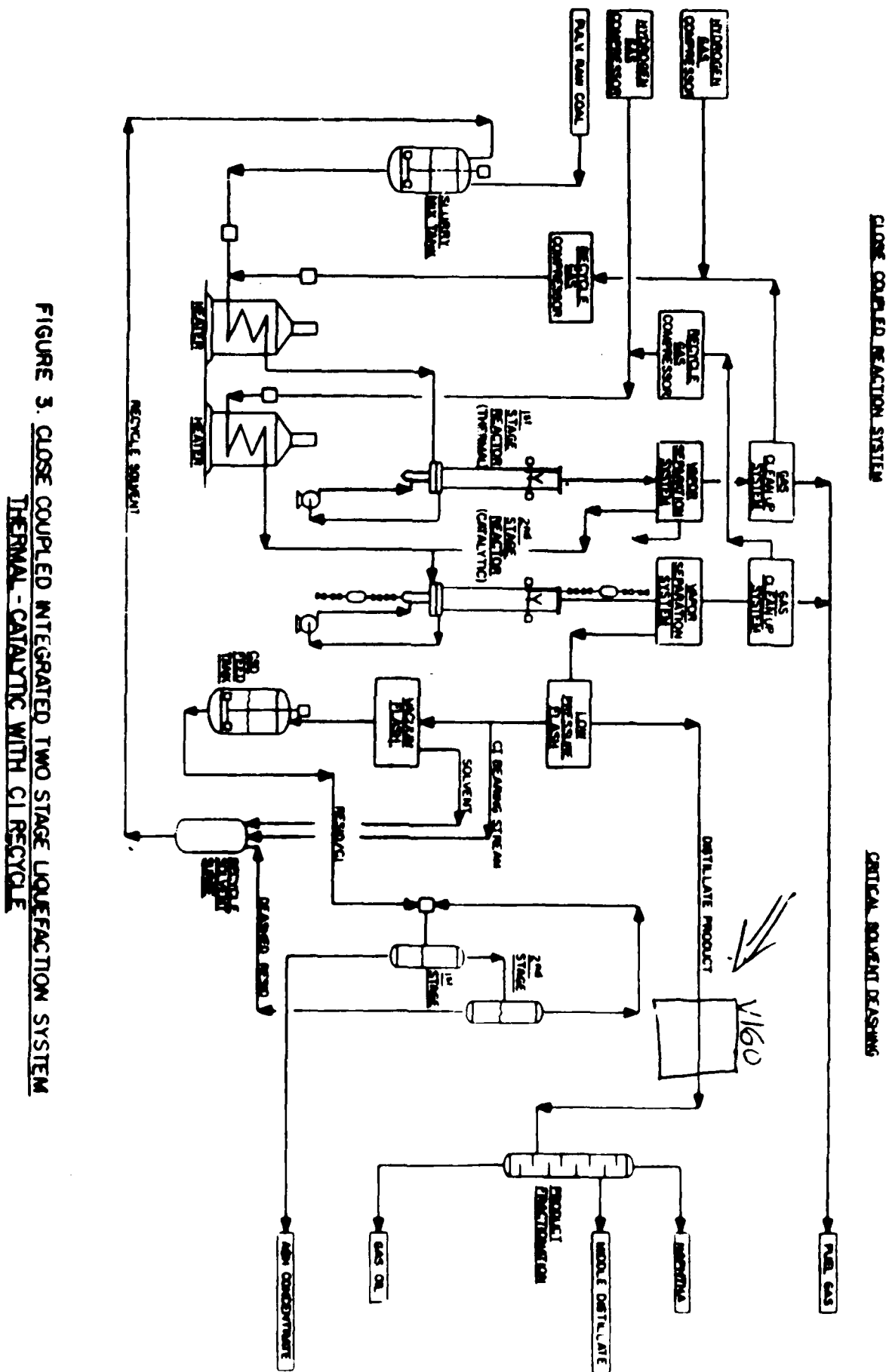
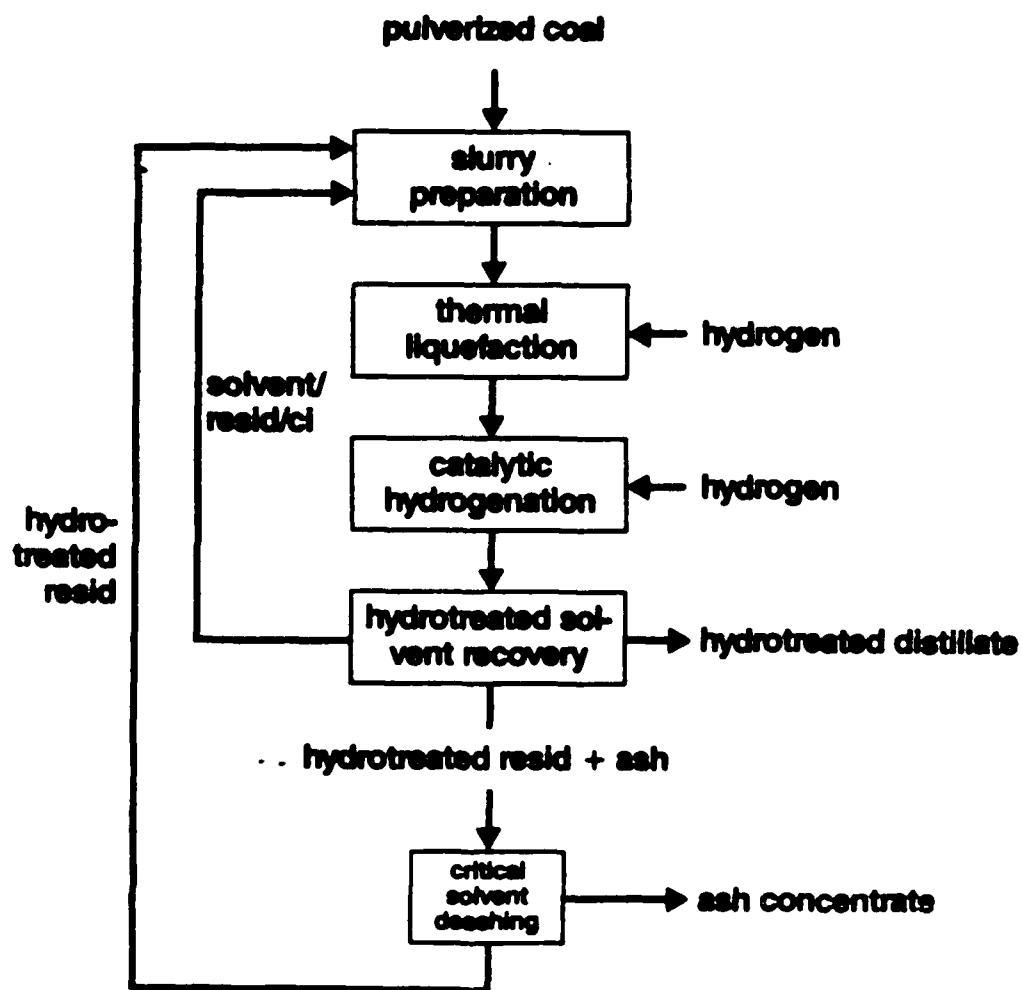


FIGURE 3. CLOSE COUPLED INTEGRATED TWO STAGE LIQUEFACTION SYSTEM
THERMAL - CATALYTIC WITH CI RECYCLE

**CC-ITSL with ash recycle
two-stage liquefaction
close-coupled mode**



9175-71

CATALYTIC



J&A ASSOCIATES, INC.

18200 West Highway 72 Golden, Colorado 80401

(303) 425-6021

October 27, 1986

Mr. John Downen

AFWAL/POSP

Wright Patterson Air Force Base
OHIO 45433-6563

Subject: Preparation of Coal Liquid
Hydrotreater Feed

Reference: J&A Data Report #
86-10-582-3510

Dear John,

As you requested in your P. O. # WL1, we have distilled two drums of Subbituminous Coal Liquid to your specifications in order to prepare a feedstock for hydrotreating tests. The drums were received from Catalytic, Inc. Wilsonville, Alabama on August 29, 1986 and the desired distillate was shipped to Wright-Patterson AFB on ~~September~~ ^{October} 15, 1986. The feedstock produced consisted of 61.3 gallons of 26.6° API oil.

The distillation equipment used was our 25 gallon Batch Still (Figure 3) which was run at atmospheric pressure until the kettle reached 550°F and then under vacuum to take the 625°F cut (approximately 500°F max kettle temperature). This still takes cuts similar to those obtained from a True Boiling Point (TBP) distillation, ASTM D 2892. Each run of 25 gallons took two (2) days, even using a reflux ratio of 1:1 in order to speed up the work. During the distillations, data were recorded each time a receiver was drained (3.5 liter). Products were nitrogen blanketed as they were collected in order to preclude oxidation and polymerization.

Results are given in the enclosed tables and figures.

Table 1 gives the overall results and the analyses requested on the 300-625°F fraction (hydrotreater feedstock).

Tables 2 - 5 are the distillation reports giving weights, volumes, recoveries, and readings from the four (4) runs performed.

Figure 1 is a composite distillation graph, showing points from all four (4) runs.

Figure 2 is a plot of the product (300-625°F) density versus temperature, as requested by Captain William Harrison III.

Results obtained agreed reasonably well with the Catalytic, Inc. simulated distillation which predicted a 55.5 weight percent yield of 300-625°F, versus our result of 59.0 weight percent. Our initial D86 distillation indicated a 53% yield, but this distillation was run very slowly due to intense foaming of the sample. Therefore the D86 result on the whole oil is not reported here and should be discarded if you have a copy.



Page 2

The IBP-300°F and 625°F+ fraction have been retained. Please advise us as to their disposal. Small samples of the whole oil and the 300-625°F fraction have also been retained if you need more analyses performed.

If you have any questions about the results or procedures, please call me or Dr. Mark Atwood at 303-425-6021.

Very truly yours,

C M Smits

CARL M. SMITS
Laboratory Supervisor

CMS/eac

Encs.

cc: Mr. Dennis Morrell,	Hercules Res.
Mr. Timothy Dues,	WPAFB
Captain William Harrison III,	WPAFB
Dr. Mark T. Atwood,	J&A Associates
File Copy	

Table 1

OVERALL RESULTS ON TWO DRUMS OF SUBBITUMINOUS COAL LIQUID

<u>J&A Sample #</u>	<u>#56503-A</u>	<u>#56503-B</u>	<u>#56303-C</u>	
<u>TBP Fraction</u>	<u>IBP-300°F</u>	<u>300-625°F</u>	<u>625°F+</u>	<u>Totals</u>
Weights, g	50,330	207,780	94,355	352,465
Wt % of charge	14.29	58.98	26.78	100.05
Volumes, mls at 60°F	66,990	232,098	98,178	397,266
Volumes, gallon at 60°F	17.7	61.3	25.9	104.9
Volume percent of charge	16.89	58.52	24.75	100.16
API Gravity, 60/60	56.9 ^o	26.6 ^o	15.7 ^o	27.8 ^o
Specific Gravity	0.7513	0.8952	0.9611	0.8882
Elemental Analysis, wt %				
Carbon (PE 240C)		86.83		
Hydrogen (PE 240C)		12.84		
Nitrogen (Antek)		0.25		
Sulfur (XRF)		0.01 (131 wppm)		
Oxygen (by difference)		0.07		
D86 Distillation, °F at 1 atmos				
IBP/5 vol % over		340/360		
10/20 vol % over		382/409		
30/40		437/466		
50/60		486/506		
70/80		524/544		
90/95		566/581		
EP, °F/EP, Vol % over		590/98.5		
% Residue		0.5		
% Recovery		99.0		

Table 2

TBP DISTILLATION REPORT

Dist Type	TBP	Dist #	1 of 4	Analyst	C. M. Smits
JAA Number	56	Proj #	3510	Date	October 2, 1986
Sample Description	n 2 Drums Subbituminous Coal Liquid Received 9/29/86 from Catalytic, Inc.				

Cut Number	Temp. Range F at 1 Atm.	Weight (gms)	Wt %	Σ Wt %	Specific Gravity	°API 60/60	Volume (ml)	Volume Percent	Σ Volume Percent
56503-A	IBP(72)-300	12,075	14.57	14.57	0.7544	56.1	16,006	17.15	17.15
56503-B	300-625	48,590	58.62	73.19	0.8954	26.5	54,266	58.15	75.30
	Total Distilled	60,665	73.19				70,272	75.30	
56503-C	625+	22,380	27.00	100.19	0.9612	15.7	23,283	24.95	100.25
	Totals	83,045					93,555		

Vol % Over	Overhead Temp. °F	Vol % over	Overhead Temp. °F
0.4	72 (IBP)	40.4	480
4.1	179	44.2	499
7.8	208	47.9	518
11.6	244	51.7	533
15.3	281	55.4	548
17.2	300	59.2	566
20.9	332	62.9	575
24.7	362	66.7	583
28.4	384	70.4	600
32.9	432	74.2	620
36.7	456	75.3	625

Actual Balance Data:

Charge:	82,885	gms.	93,318	gms.	SG =	82,885
Recovery:	83,045	gms.	93,555	gms.	SG =	.8882
Loss + Hold-Up:	(160)	gms.	(237)	gms.	SG =	22,380
% Recovery:	100.19	Wt %	100.25	Wt %	SG =	.9612
					API =	27.8°
					API =	15.7°

Disk: Form
Doc: JAA0014
10/15/86

Table 3

TBP DISTILLATION REPORT

Dist Type	TBP	Dist #	2 of 4	Analyst	C. M. Smits				
J&A Number	56503	Proj #	3510	Date	October 4, 1986				
Sample Description	2 Drums Subbituminous Coal Liquid Received 9/29/86 from Catalytic, Inc.								
Cut Number	Temp. Range F at 1 Atm.	Weight (gm)	Wt %	Σ Wt %	Specific Gravity	°API 60/60	Volume (ml)	Volume Percent	Σ Volume Percent
56503-A	IBP(73)-300	13,505	14.18	14.18	0.7540	56.2	17.911	16.70	16.70
56503-B	300-625	58,110	61.01	75.19	0.8973	26.2	64,761	60.39	77.09
	Total Distilled	71,615	75.19				82,672	77.09	
56503-C	625+	23,595	24.77	99.96	0.9622	15.6	24,522	22.87	99.96
	Totals	95,210					107,194		

Vol % Over	Overhead Temp. °F	Vol % over	Overhead Temp. °F	Vol. % Over	Overhead Temp. °F
0.2	73 (IBP)	36.5	446	72.5	601
3.4	173	39.9	468	75.8	617
6.7	206	43.2	482	77.1	625
10.0	237	46.5	495		
13.2	266	49.7	512		
16.7	300	53.0	520		
20.1	340	56.2	532		
23.3	352	59.5	545		
26.6	376	62.8	563		
29.8	399	66.0	574		
33.1	424	69.3	587		

NOTE: This distillation experienced a small vacuum leak. It is believed the recorded temperatures above 500°F should be approximately 10°F higher.

Actual Balance Data:

Charge:	95.250	gms.	107.239	mls.	Charge Data:	SG = 95.250
Recovery:	95.210	gms.	107.194	mls.		SG = .8982
Loss + Hold-Up:	40	gms.	45	mls.	Residuum Data:	SG = 23.595
% Recovery:	99.96	Wt %	99.96	Vol %		SG = .9622
						API = 15.6°

Disk: Form
Doc: JAA0014
10/15/86

Table 4

TBP DISTILLATION REPORT

Dist Type	TBP	Dist #	3 of 4	Analyst	C. M. Smits				
J&A Number	56503	Proj #	3510	Date	October 9, 1986				
Sample Description	2 Drums Subbituminous Coal Liquid Received 9/29/86 from Catalytic, Inc.								
Cut Number	Temp. Range F at 1 Atm.	Weight (gms)	Wt %	Σ Wt %	Specific Gravity	$^{\circ}$ API 60/60	Volume (ml)	Volume Percent	Σ Volume Percent
56503-A	IBP(71)-300	13,340	14.36	14.36	0.7460	58.2	17,882	17.10	17.10
56503-B	300-625	53,655	57.77	72.13	0.8940	26.8	60,017	57.39	74.49
	Total Distilled	66,995	72.13				77,899	74.49	
56503-C	625+	25,995	27.99	100.12	0.9603	15.9	27,070	25.89	100.38
	Totals	92,990					104,969		

Vol % Over	Overhead Temp. $^{\circ}$ F	Vol % over	Overhead Temp. $^{\circ}$ F	Vol. % Over	Overhead Temp. $^{\circ}$ F
0.5	71 (IBP)	35.9	451	71.5	615
3.9	179	39.2	472	74.5	625
7.2	209	42.4	488		
10.6	241	46.4	500		
14.0	273	49.5	526		
17.1	300	52.7	543		
20.2	330	55.8	550		
23.4	357	59.0	567		
26.5	378	62.1	580		
29.7	405	65.2	589		
32.9	430	68.4	600		

Actual Balance Data:					
Charge:	92,880	gms.	104,571	mls.	Charge Data: SG = 92,880
Recovery:	92,990	gms.	104,969	mls.	SG = .8882 API = 27.8 $^{\circ}$
Loss + Hold-Up:	(110)	gms.	(398)	mls.	Residuum Data: SG = 25,995
% Recovery:	100.12	Wt %	100.38	Vol %	SG = .9603 API = 15.9 $^{\circ}$

Disk: Form
Doc: JAA0014
10/15/86

Table 5

TBP DISTILLATION REPORT

Dist Type	TBP	Dist #	4 of 4	Analyst	C. M. Smits				
J&A Number	56503	Proj #	3510	Date	October 13, 1986				
Sample Description	2 Drums Subbituminous Coal Liquid Received 9/29/86 from Catalytic, Inc.								
Cut Number	Temp. Range F at 1 Atm.	Weight (gms)	Wt %	Σ Wt %	Specific Gravity	$^{\circ}$ API 60/60	Volume (ml)	Volume Percent	Σ Volume Percent
56503-A	IBP(83)-300	11,410	14.04	14.04	0.7511	56.9	15,191	16.60	16.60
56503-B	300-625	47,425	58.35	72.39	0.8939	26.8	53,054	57.98	74.58
	Total Distilled	58,835	72.39				68,245	74.58	
56503-C	625+	22,385	27.54	99.93	0.9606	15.8	23,303	25.47	100.05
	Totals	81,220					91,548		
Vol % Over	Overhead Temp. $^{\circ}$ F		Vol % over		Overhead Temp. $^{\circ}$ F				
0.5	83 (IBP)								
4.2	181		36.1		445				
7.9	215		40.5		480				
11.7	251		44.3		495				
15.4	290		48.1		514				
16.6	300		51.9		530				
20.4	332		55.7		539				
24.3	362		59.6		552				
28.1	390		63.4		564				
31.9	421		67.2		590				
			71.0		609				
			74.6		625				

Actual Balance Data:

Charge:	81,275	gms.	91,505	mls.	Charge Data:	SG =	81,275	API =	27.8 $^{\circ}$
Recovery:	81,220	gms.	91,548	mls.		SG =	.8882	API =	
Loss + Hold-Up:	55	gms.	(43)	mls.	Residuum Data:	SG =	22,385	API =	
% Recovery:	99.93	Wt %	100.05	Vol %		SG =	.9606	API =	15.8 $^{\circ}$

Disk: Form
Doc: JAA0014
10/15/86

FIG. 1

SUBJECT: SUBBITUMINOUS COAL LIQUID-VI60 PAGE _____ OF _____

FROM CATALYTIC, INC. DATE: REC'D 9/29/86 PROJECT # _____

REF # 56503 ANALYST: C M Smith

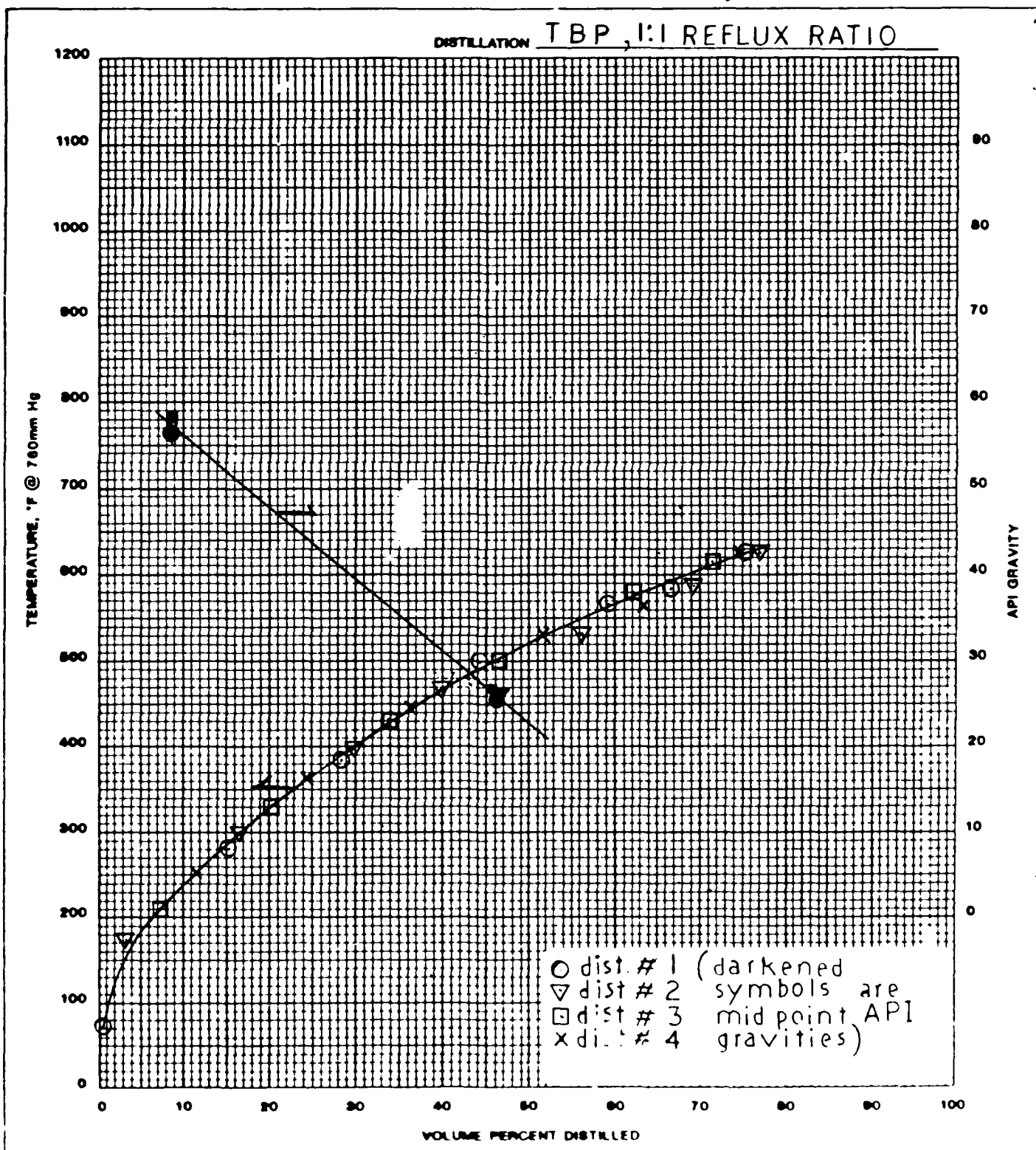
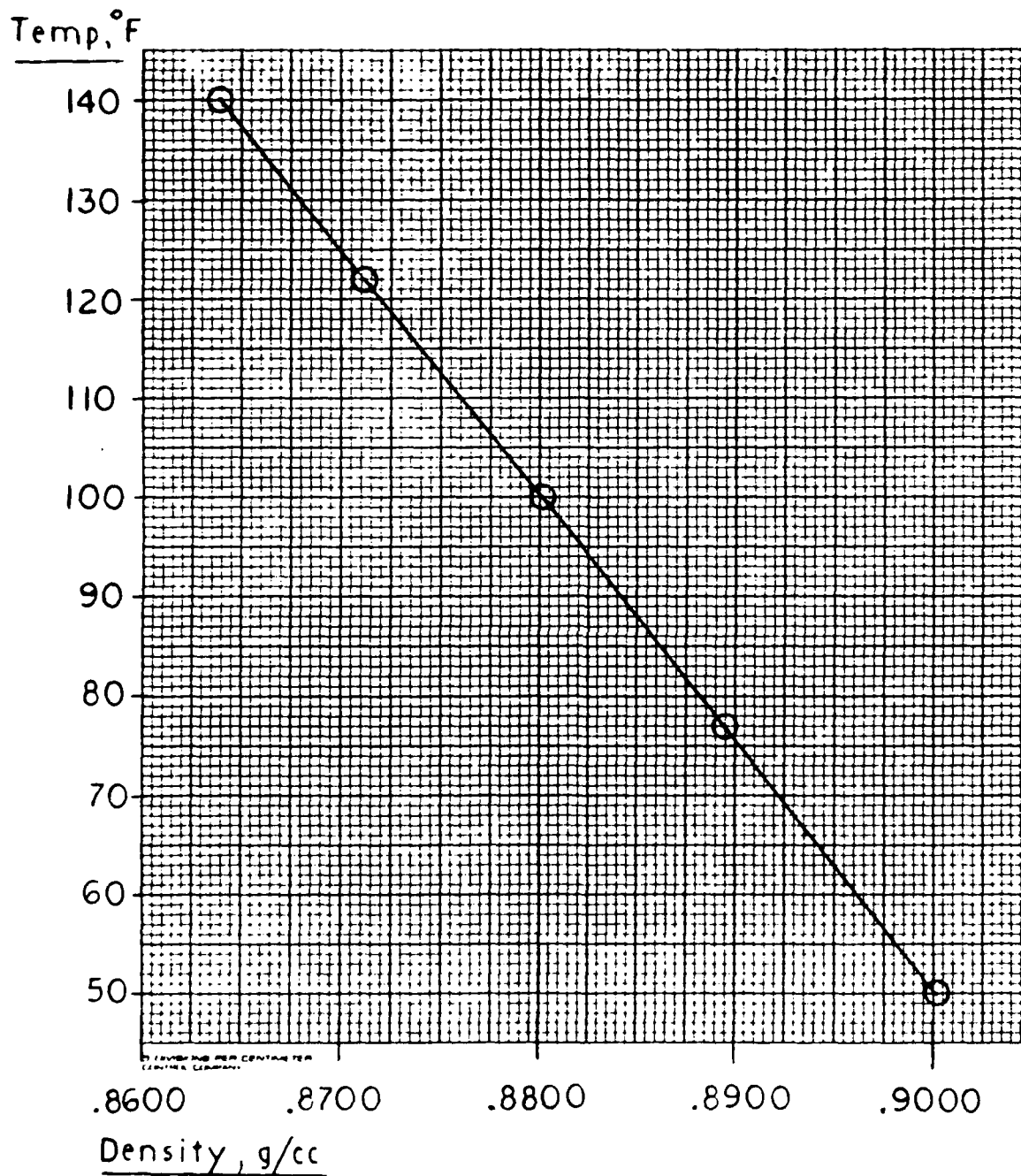


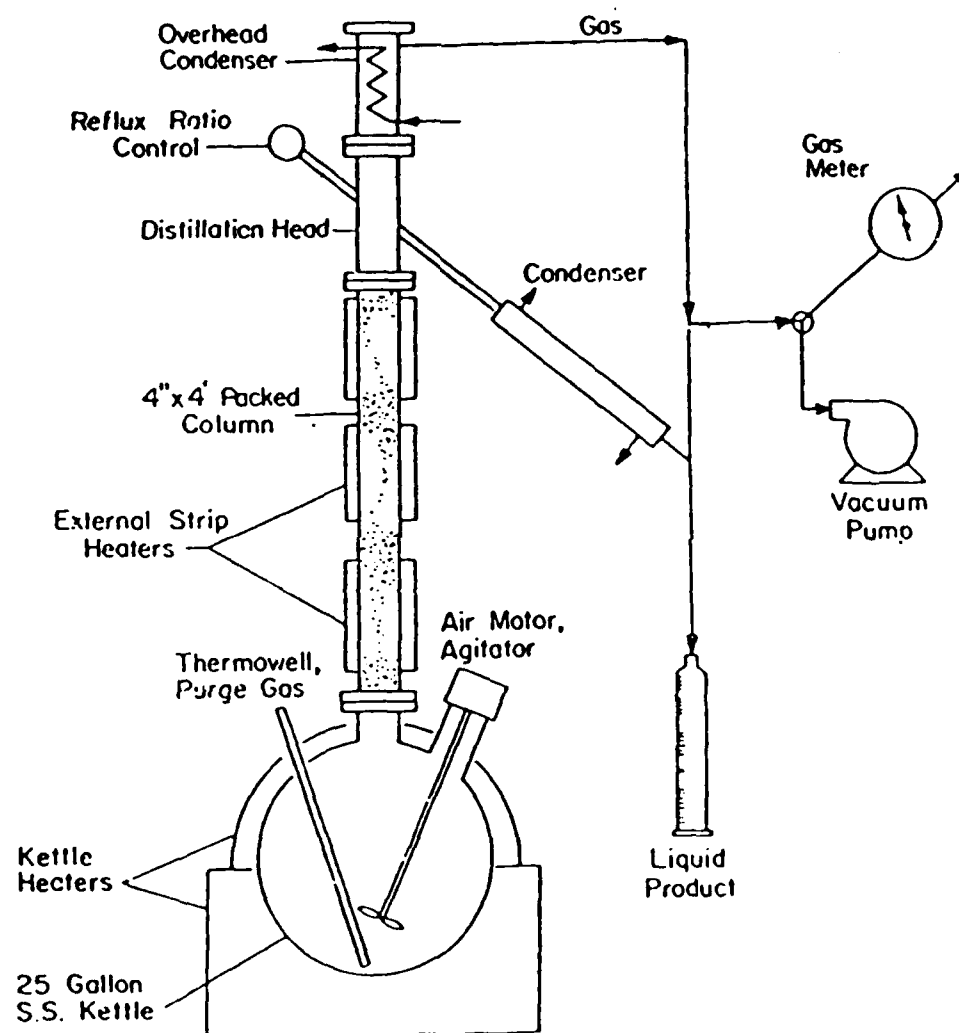
Figure 2

DENSITY VERSUS TEMPERATURE, 300-625°F FRACTION
SUBBITUMINOUS COAL LIQUID

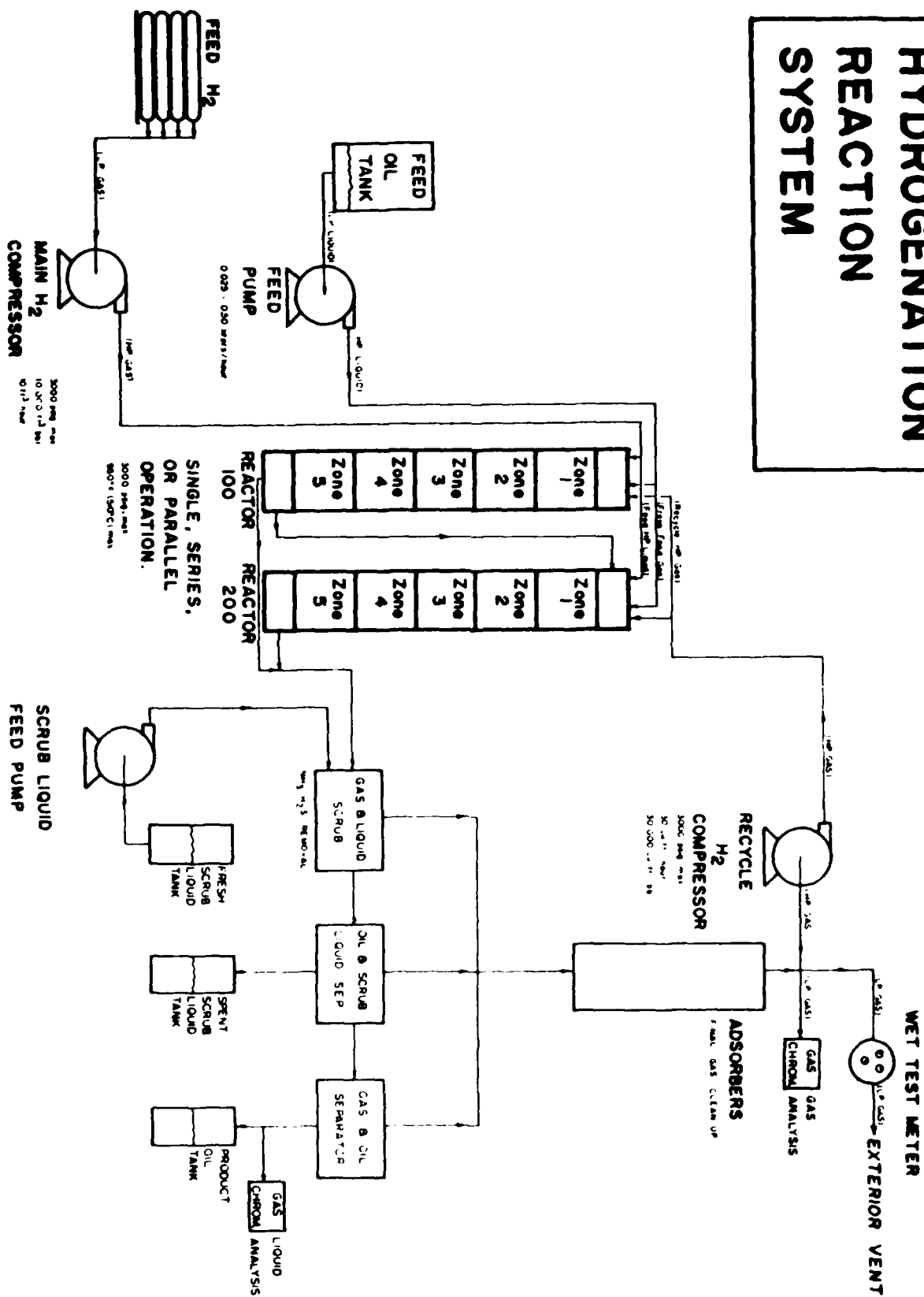


<u>°F</u>	<u>°C</u>	<u>Density, g/cc</u>
50	10	0.9002
77	25	0.8895
100	37.8	0.8802
122	50	0.8712
140	60	0.8639

FIG. 3
25 GALLON BATCH STILL



HYDROGENATION REACTION SYSTEM



COURTESY OF AFWAL/POSF



Shell 424 Ni/Mo Hydrotreating Catalyst

Introduction

Shell 424 is a third generation nickel molybdenum catalyst on an extruded alumina base. It was developed for use in severe, heavy oil services in which diffusional limitations may be a problem. This applies to all heavier than distillate feedstocks. Shell 424 provides much better activity and stability than traditional Ni-Mo catalysts. This catalyst differs from Shell 324 in several ways

- The most significant change is a totally new technology for distributing the metals on the catalyst. The technique provides a much more even dispersion of metals throughout the catalyst.
- The base has been modified to make the catalyst more tolerant to metals and contaminants.
- Shell 424 was the first shaped catalyst Shell produced commercially. The tri-lobed shape also reduces diffusional limitations. This catalyst has the same high strength characteristics that all Shell catalysts share, and the product has been dense loaded in nearly every application to date. The tri-lobed shape also provides reduced pressure drop compared with the same sized cylinder.
- The metal content was optimized for heavier feedstocks.

Typical properties

Shape	Tri-lobed	Tri-lobed	Tri-lobed
Normal size, in. (mm)	1.16 (1.5)	1.10 (2.5)	1.8 (4.0)
Chemical composition, % wt			
Nickel	3.0	3.0	4.0
Molybdenum	13.0	13.0	15.0
Physical properties			
Surface area, m ² /g	162	160	150
Pore volume, cc/g	0.47	0.47	0.47
Side plate crush strength ¹ , lb (kg)	24 (10.9)	37 (16.8)	47 (20.9)
Bulk crushing strength ² , kg/cm ²	16	17	18
Attrition index ³	99+	98+	98
Reactor loading density, lbs./ft ³ (kg/l)			
sock loaded	47-48	47-48	47-48
dense loaded	52-53	52-53	52-53
Compacted bulk density, lbs./ft ³ (kg/l)	52 (0.83)	52 (0.83)	52 (0.83)
Loss of ignition at 900° F (482° C), % wt ⁴	0.7	0.7	0.7

¹ 16 inch long particles

² Pressure applied to procedures 0.5%W fines 40 mesh

³ %Wt retained on 20 mesh screen after tumbling 1 hour at 40 rpm

⁴ As manufactured

Container weights SD 300 lb. net
BP 1500 lb. net

Commercial experience

VGO hydrotreating and hydrocracker pretreating

Shell 424 has found immediate acceptance throughout the industry. In less than one year it has been purchased by a dozen companies in over twenty locations throughout North America. It has been extremely successful in both first-stage hydrocrackers and VGO hydrotreaters. The differences in the base have also allowed refiners to put a significant amount of residue into cat. cracker pretreaters and also to treat de-asphalted oils at relatively low pressures. Shell 424 has improved stability and activity in all of these applications.

Mild hydrocracking

An additional use of Shell 424 has been in mild hydrocracking services. Several cat. cracker pretreaters are being used with Shell 424 to produce 20-30% additional distillate from VGO. The distillate has cetane indices between 40 and 45 depending on the feedstock and cracking severity. Since the cracking reaction is primarily de-alkylation of polyaromatics, the bottoms product has a very low polyaromatic content and makes a superior cat. cracker feed. Shell 424 gives this 20-30% conversion with very low production of C₄ and lighter materials.

Distillate hydrotreating

Shell 424 is in service treating coker and cracked distillates. The high hydrogenation, denitrification activity will yield improvement in product quality at high severity operations.

Packing and availability

Shell 424 is available as 1/16, 1/10, and 1/8 inch extrudates packed at 300 lbs net weight in 55 gallon steel drums or 1500 lbs net weight in sling bins. Orders normally can be filled on short notice from inventories maintained at Shell's West Coast manufacturing plant. Small inventories are typically kept on the Gulf Coast for emergency or top up needs.

Health, safety, and environmental precautions

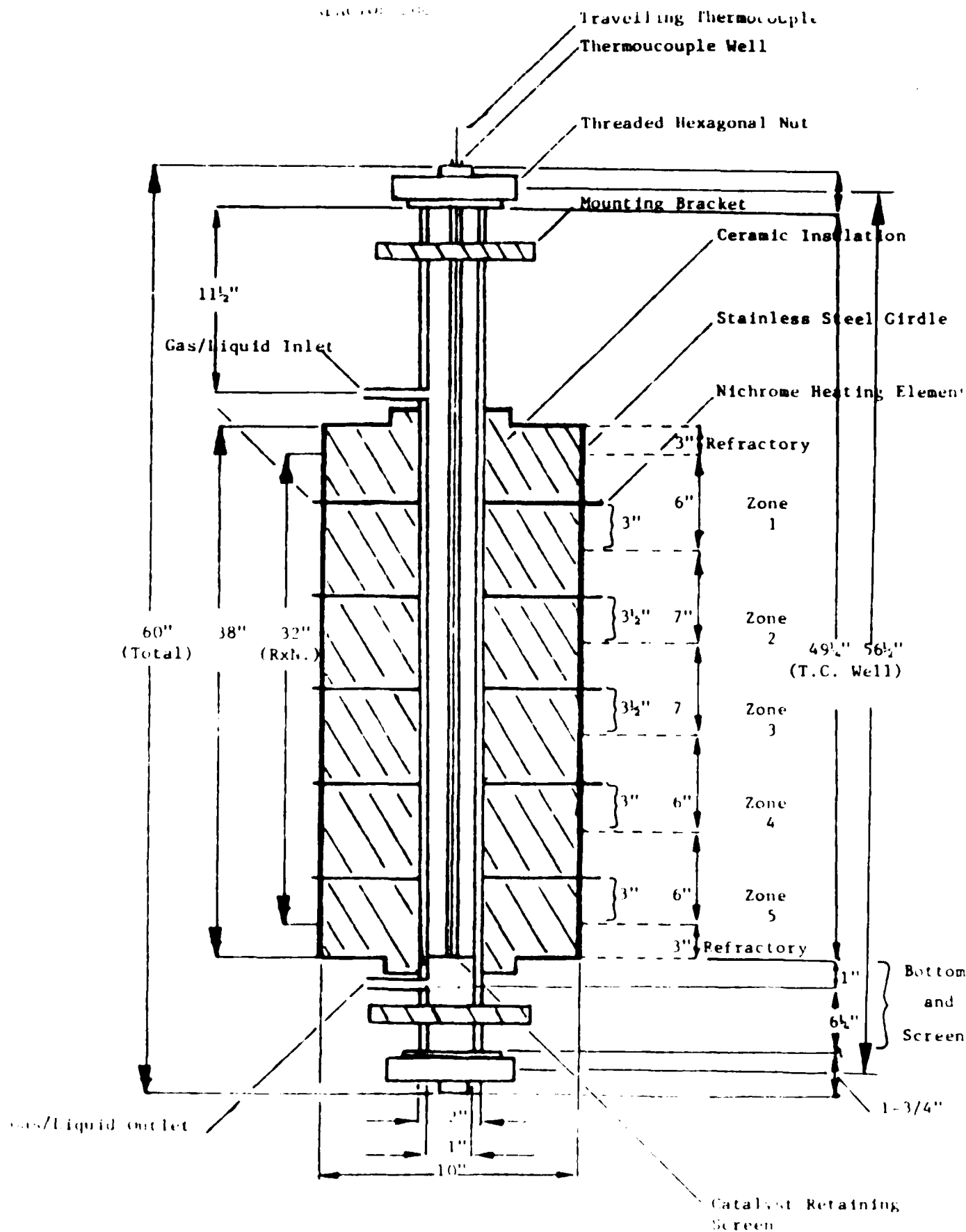
Shell 424 Catalyst is made from chemicals which span a range from being practically nontoxic to being potential carcinogens. Full attention to these hazards, and to appropriate precautions and preventative measures is essential. Before ordering, testing or using these catalysts, available information on health, safety, and environmental hazards, precautions, and preventative measures must be obtained from your Shell Chemical Catalyst Sales Representative.

For additional information, call or write:

Shell Chemical Company
Catalyst Business Center
One Shell Plaza
P.O. Box 2463
Houston, Texas 77252
Telephone (713) 241-4997 or 241-4927

Warranty

All products purchased from Shell are subject to terms and conditions set out in the contract, order acknowledgment and/or bill of lading. Shell warrants only that its product will meet those specifications designated as such herein or in other publications. All other information supplied by Shell is considered accurate but is furnished upon the express condition that the customer shall make its own assessment to determine the product's suitability for a particular purpose. No warranty is expressed or implied regarding such other information, the data upon which the same is based, or the results to be obtained from the use thereof; that any product shall be merchantable or fit for any particular purpose; or that the use of such other information or product will not infringe any patent.

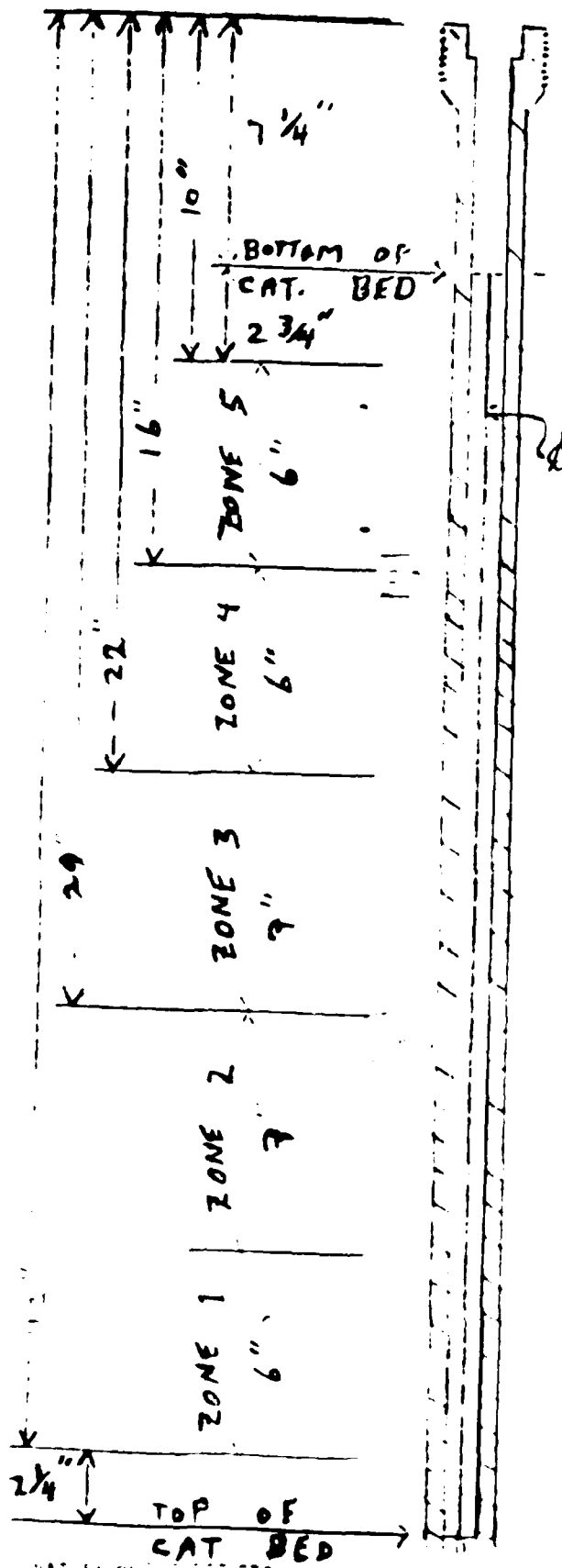


COURTESY OF AFWAL/1007
 F.

Reactor packed 2 NOV 86
 Jackson/Schneider
 as packed
 measurement

Reactor 200 packing

— 7 1/4



8 3/4" Alumina B. 113 cc
 Sand 38 cc

2" Sand

— 15 1/2

— 20

18" 200 cc Catalyst STELL 424
 32 cc Sand of 11 Mo

Pack 10 cc catalyst
 2 cc sand

2" OTTAWA SAND 26 cc

— 30 1/4

— 35 1/4

6 1/4" Alumina B. 113 cc
 Sand 27 cc

— 44 1/4

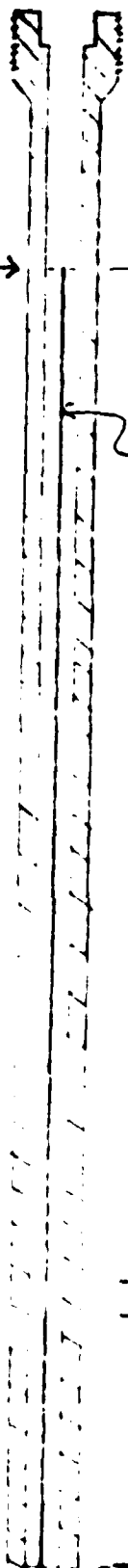
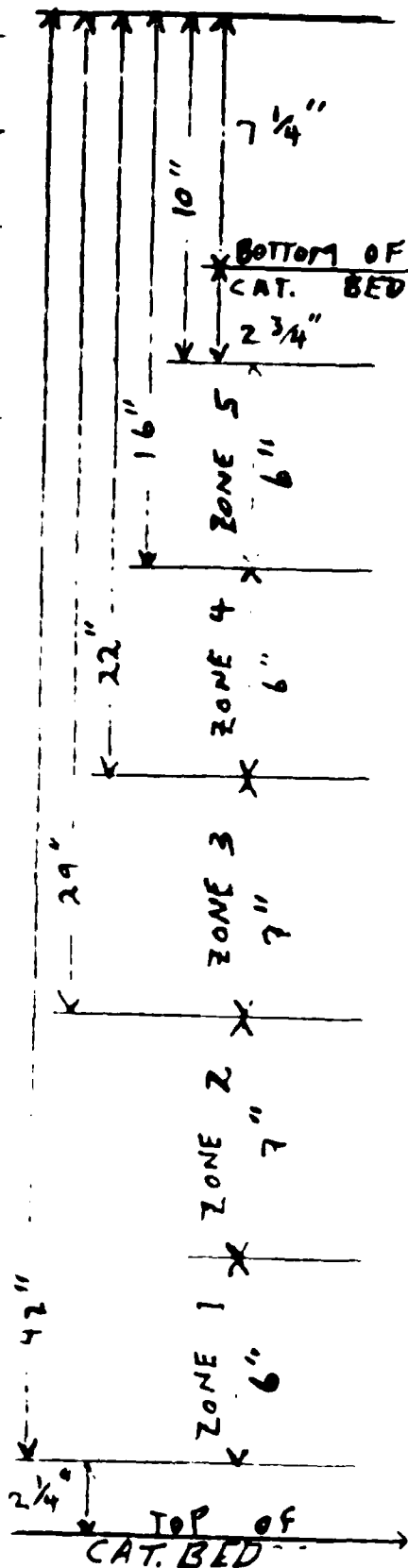
TOP OF
 CAT BED

DATE
 3 NOV 86

SUBJECT

Urethane Enclosed
11/28/86
Schumacher and
Hogden
PAGE 1
copy
mailed

RECTOR 200 PROCK 1:10
SCHEME
JD-2 CATAL LIGANDS
SBIC



2 3/4" OTAWA SAND

CATALYST ZONE

150 cc catalyst
150 cc alumina balls
150 cc sand

HERCULEY 0.5W-10
PLATINUM ON CARBON
(PRE-SULFIDED)

Feeded See catalyst
See column below
See sand
Feeded (See above) See catalyst in
See (See above) in mixture
Mixture (See above) to feed and sand
Catalyst (See above) to feed
(See above) mixture (See above)

CATALYST SEE ABOVE
0.0" to 25.0"

2" OTAWA SAND

6 1/4" ALUMINA BALLS 81cc
SAND 270cc

— 71
— 10

— 30
— 35

— 44

SIGNATURE OF PROJECT OFFICER
W. Schumacher
SIGNATURE OF WITNESS
SIGNATURE OF WITNESS

28 NOV 86

A P P E N D I X C

TEST METHODS

6 Feb 1987

John:

1. Here's a list of the test methods that were used to analyse the fuel samples for your program:

PONA	ASTM-D-2789 (Pratt & Whitney)
Distillation	ASTM-D-2887 (SFTLA)
Ref. Index	Reichert ABBE MARK 11 (POSF)
Total Aromatics	*
Secific Gravity	ASTM-D-1298 (SFTLA)
Hydrogen Content	NMR (POSF)
Nitrogen Content	Antec Nitrogen Analyser (POSF)
Sulfur Content	Horiba Sulfur Analyser (SFTLA)
GC	(POSF)
GC/MS	(POSF)
Net Heat of Comb.	ASTM-D-3338 (SFTLA)
Thermal Stability	ASTM-D-3241 (SFTLA)

* Reference "HYDROCARBON GROUP TYPE ANALYSER SYSTEM FOR THE RAPID DETERMINATION OF SATURATES, OLEFINS, AND AROMATICS ON HYDROCARBON DISTILLATE PRODUCTS" by Hayes and Anderson, Analytical Chemistry, November 1985.

() Indicates who did the actual analysis


Bob Morris

AW-PLC-EFTLA (J. YOUNG)

DATE _____

PLEASE PERFORM THE FOLLOWING SPECIFICATION TESTS AND FORWARD THE DATA TO CAPT. ~~BOB MOORE~~ ^{BOB MOORE}, AFMIL/POSF, BLDG. 499, ROOM 154, BMT. 53020. ANY QUESTIONS REGARDING THE PROPERTIES OF THIS SAMPLE CAN BE DIRECTED TO ~~BOB MOORE~~ OR MIKE SCHUMACHER AT 53020. THANK YOU FOR YOUR SUPPORT AND COOPERATION.

SIGNED Robert W. Moore

Wm:

FYI

BM

TEST SPECIFICATION TESTS TO BE DONE

NAME NUMBER PROPERTY COMMENTS

APPEARANCE

0.0001 COLOR (DENSITY/TRANSPARENCY)

COMPOSITION

0.0002 TOTAL ACID NUMBER (mg KOH/g)

0.0003 APPEARANCE (COLOR, CLARITY, VISCOSITY)

0.0004 FORMALIN (SOLUBLE AT 20)

0.0005 FORMALIN (SOLUBLE AT 20)

COMPOSITION

0.0006 ANALYSIS (ANALYSIS) PLEASE PROVIDE PRINTOUT AND ANALYSIS FOR FORMALIN INFORMATION THAN FORMALIN INFORMATION

0.0007 ANALYSIS (ANALYSIS) TO INCLUDE INITIAL FORMALIN ANALYSIS AND FORMALIN ANALYSIS AT EACH 10% RECOVERY, END POINT, AND FORMALIN

0.0008 FORMALIN (SOLUBLE AT 20)

0.0009 FORMALIN (SOLUBLE AT 20) USED FORMALIN (SOLUBLE AT 20)

0.0010 FORMALIN (SOLUBLE AT 20) USED FORMALIN (SOLUBLE AT 20)

0.0011 FORMALIN (SOLUBLE AT 20) USED FORMALIN (SOLUBLE AT 20)

0.0012 FORMALIN (SOLUBLE AT 20)

0.0013 FORMALIN (SOLUBLE AT 20)

0.0014 FORMALIN (SOLUBLE AT 20)

0.0015 FORMALIN (SOLUBLE AT 20) CALCULATE

0.0016 FORMALIN (SOLUBLE AT 20)

0.0017 FORMALIN (SOLUBLE AT 20) DEGREES F

0.0018 FORMALIN (SOLUBLE AT 20) DEGREES C, OR 30 DEGREES F

0.0019 FORMALIN (SOLUBLE AT 20)

0.0020 FORMALIN (SOLUBLE AT 20)

COMBUSTION

0.0021 ANILINE-DENSITY PRODUCT

0.0022 NET HEAT OF COMBUSTION

0.0023 NET HEAT OF COMBUSTION, HIGH PRECISION

0.0024 ESTIMATION OF NET HEAT OF COMBUSTION, USE 0.0023 DATA

0.0025 COUNDMETER NUMBER

0.0026 SMOKE POINT, MM

0.0027 ESTIMATION OF HYDROGEN CONTENT, USE 0.0024 DATA

0.0028 NAPHTHALENES

CORROSION

0.0029 COPPER STRIP CORROSION, 2 HR AT 100C

STABILITY

0.0030 STABILITY (STABILITY) 100% 100% 100% 100%

CONTAMINANTS

0.0031 ESTIMATION OF 100% 100%

0.0032 FORMALIN (SOLUBLE AT 20) AND FORMALIN (SOLUBLE AT 20)

0.0033 WATER REACTION (SOLUBLE AT 20) AND FORMALIN (SOLUBLE AT 20)

0.0034 WATER REACTION (SOLUBLE AT 20) AND FORMALIN (SOLUBLE AT 20)

0.0035 FORMALIN (SOLUBLE AT 20) AND FORMALIN (SOLUBLE AT 20)

OTHER TESTS

0.0036 FORMALIN (SOLUBLE AT 20) AND FORMALIN (SOLUBLE AT 20)

0.0037 FORMALIN (SOLUBLE AT 20)

0.0038 FORMALIN (SOLUBLE AT 20)

0.0039 FORMALIN (SOLUBLE AT 20)

0.0040 FORMALIN (SOLUBLE AT 20) AND FORMALIN (SOLUBLE AT 20)

0.0041 FORMALIN (SOLUBLE AT 20)

0.0042 FORMALIN (SOLUBLE AT 20)

Test Data

1	WT. % N 2	WT. % S 3	WT. % H 4	Density 5	% Aromatics 6
HRS-001	NR	NR	NR	NR	NR
HRS-002	NR	0.1384	9.171	0.9464	NR
HRS-003	0.1998	NR	NR	0.9249	NR
HRS-004	1.6720	NR	NR	0.9563	NR
HRS-005	0.005301	NR	NR	0.9215	NR
HRS-006	0.02297	0.1035	9.972	0.9279	NR
HRS-007	0.007144	0.1086	9.876	0.9236	NR
HRS-008	0.003755	0.0469	12.379	0.8607	NR
HRS-009	0.03493	NR	7.136	1.0152	NR
HRS-010	0.0004286	NR	NR	0.9240	NR
HRS-011	0.0003010	*	*	0.9-02	NR
HRS-012	0.000043	0.0549	12.292	0.8634	NR
HRS-013	0.00007703	0.0477	12.769	0.8339	NR
HRS-014	0.02407	0.2184	9.813	0.9339	NR
HRS-015	0.0104	0.0567	12.442	0.8430	NR
HRS-016	0.2975	0.0433	12.010	0.8902	
HRS-017	0.0737	0.0426	12.450	0.8767	
HRS-018	0.06843	0.0377	12.505	0.8776	
HRS-019	0.07381	0.0425	12.479	0.8778	
HRS-020	0.001898	0.0329	12.933	0.8675	
HRS-021	0.003608	*	12.755	0.8693	
FEED HRS-022	0.2928	0.0509	12.071	0.8901	
HRS-023	0.006227	0.0344	12.771	0.8692	
HRS-024	0.006249	0.0343	12.790	0.8694	
HRS-025	0.007211	0.0363	12.733	0.8687	
FEED HRS-026	0.007-33	0.0358	12.776	0.8697	
FEED HRS-027	0.007566	0.0354	12.691	0.8700	
HRS-028	0.005275	0.0286	12.926	0.8644	
HRS-029	0.007217	0.0291	12.701	0.8700	
HRS-030	0.004732	0.0292	12.943	0.8661	
HRS-031	0.1416	0.0322	12.513	0.8316	
HRS-032	0.1405	0.0401	12.290	0.8315	
HRS-033	0.1367	0.0413	12.294	0.8315	
HRS-034	0.2613	0.0401	12.290	0.8312	
HRS-035	0.164	0.0332	12.571	0.8366	
HRS-036	0.1514	0.0404	12.594	0.8276	

END

8-87

DTIC