

MASTER

CATALYSTS FOR UPGRADING COAL-DERIVED LIQUIDS

Quarterly Report for the Period
October 1 - December 31, 1980

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CATALYSTS FOR UP-GRADING COAL DERIVED LIQUIDS
(DE-14876-5)

ABSTRACT

A linear relationship represents the hydrogenation activity decay of catalysts used in four experimental runs reported previously. The weight percent hydrogen in the reactor product oils plotted against oil-catalyst contact time for experimental runs ZBB, ZBC, ZBD and ZBE reveals a linear decay rate of 0.0083 wt% hydrogen per hour. This is one quantitative measure of catalyst activity decay. The data for the plot incorporate three different catalysts or combinations used to process a PAMCO liquid at 1500 psig, 435C and LVHST of 2 hours. The data set covers run duration of up to 120 hours of oil-catalyst contact.

An air driven hydrogen compressor was installed in the Catalyst Life Test Unit to reduce the costs associated with bottle hydrogen. Minor repairs were made on the oil feed pump.

Five experimental runs were made with Shell 324 NiMo/Al catalyst using two feedstocks: 1) 40 wt% EDS/EDS raw solvent and 2) 30 wt% SRC-I/creosote oil. The EDS feed oil proved to be rather easily hydro-treated as evidenced by 82-100% nitrogen removal, essentially complete desulfurization and no catalyst activity decay during 260 hours of continuous operation. Rapid coking resulted from the highly hydrogen deficient SRC/creosote mixture.

The Shell 324 catalyst gave excellent hydrogenation of both liquids by increasing the product oils hydrogen content by about 3.8 wt%. This catalyst will be used in future studies; however, a new feedstock consisting of 30 wt% SRC-I/PAMCO process solvent will be assessed for use in catalyst decay mechanism studies.

OBJECTIVE AND SCOPE OF WORK

The goal of this program is to investigate catalysts for upgrading liquids derived from coal-to-oil processes.

This research has the following technical objectives:

1. Investigate mechanisms responsible for rapid initial catalyst deactivation.
2. Study use of "throw away" catalysts and staged catalyst beds in hydroprocessing of coal liquids.

The investigation shall consist of the following tasks:

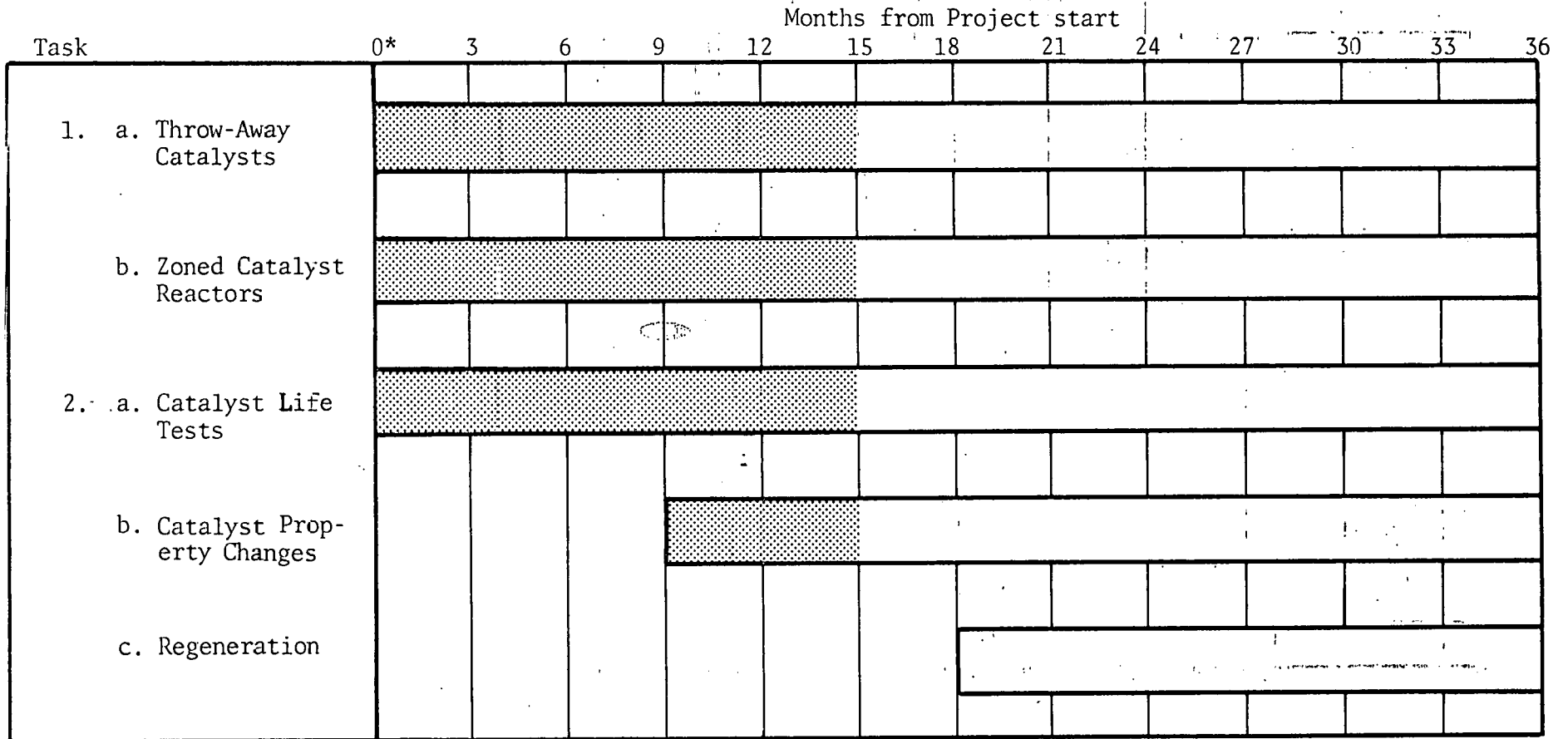
- Task 1-a. Inexpensive "throw away" type catalyst for coal liquids upgrading shall be investigated for reactor guard chamber service.
- Task 1-b. Reactors packed with mixtures of catalyst types and zoned catalyst configurations shall be evaluated.
- Task 2-a. Catalyst life tests shall be conducted using a standard Ni-Mo/alumina hydroprocessing catalyst in the existing catalyst life test unit.
- Task 2-b. Changes in catalyst properties, including surface area, pore size distribution, pore volume, and coke and inorganics accumulation shall be measured as a function of coal-liquid contact time.
- Task 2-c. The Ni-Mo/alumina catalyst tested in Task 2-a shall be regenerated by oxidation, and tested for activity recovery.



SUMMARY OF PROGRESS TO DATE

The following chart summarizes the progress to date. The three tasks (1-a, 1-b, 2-a, and 2-b) show scheduled progress. Although not shown in the figure, some initial activity has begun in task 2-c.

As of December 31, 1980, 38% of the total budget has been expended (\$68,154). This is an expected expenditure rate for the first five quarters of this project.

PROGRESS SUMMARY AS OF DECEMBER 31, 1980



 Scheduled
 Progress

* Project start date is October 1, 1979

DESCRIPTION OF TECHNICAL PROGRESS

Task 1 - Throw Away and Zoned Catalyst Systems (O. K. Bhan)

A literature survey was conducted to establish a suitable method for determining asphaltene content in coal-derived liquids. Various analytical methods are available for asphaltene content determination, but none is standard. Others have pointed out (1,2) that no relationship exists between the asphaltenes produced by different analytical methods currently in use.

The asphaltenes have been classified by the paraffin used to precipitate them from the benzene-soluble portion of the feed. Thus, there are pentane asphaltenes, hexane asphaltenes, heptane asphaltenes, and so on (3, 4).

The analytical method for asphaltene content determination developed by Pittsburgh Energy Technology Center has been adopted for use in our laboratory.

A ten-gram sample is extracted with benzene in a Soxhlet apparatus. The benzene is removed on a steam bath, and 5 ml. of benzene are added to the residue. Asphaltenes are precipitated with 250ml of n-pentane, and separated by filtration.

Preparation of the final report (thesis) on the initial portion of this study was completed during this quarter. Most data have been presented in previous quarterly reports. Some hydrogen data not previously given are shown in Figure 1. The weight percent hydrogen in the product oil as a function of oil-catalyst contact time for experimental runs ZBB, ZBC, ZBD and ZBE(5) are presented. A steady decrease in the weight percent hydrogen in the product oil was observed for Ni-Mo-Al₂O₃ catalyst, Co-Mo-Al₂O₃ catalyst and their zonal bed combination. A linear equation was fitted to the data using a regression technique. The least squares estimate of slope is 0.00827 wt% hydrogen/hour which is one quantitative measure of catalyst activity decay for the set of experiments using different catalysts.

Different feedstocks were considered for possible use in the future zonal bed and guard chamber studies; the emphasis being on selection of high boiling liquids. Table 1 presents the boiling point data of the various feedstocks tested.

A mixture of SRC-I (solid at room temperature) and Pamco solvent will be used in future zonal catalyst bed studies.

During the coming quarter, guard chamber tests using reduced iron, manganese nodules and alumina are planned. Various new catalysts have been ordered for further study in zonal beds.

1. Schultz, H. and M. J. Mima, A.C.S., Preprints, Division of Fuel Chemistry, No. 2, Vol. 23, p. 76 (1978).
2. Schultz, H. and M. J. Mima, A.C.S., Preprints, Division of Fuel Chemistry, No. 1, Vol. 25, p. 18 (1980).
3. Long, R. B., A. C. S., Preprints, Division of Petroleum Chemistry, No. 4, Vol. 24, p. 1028 (1979).

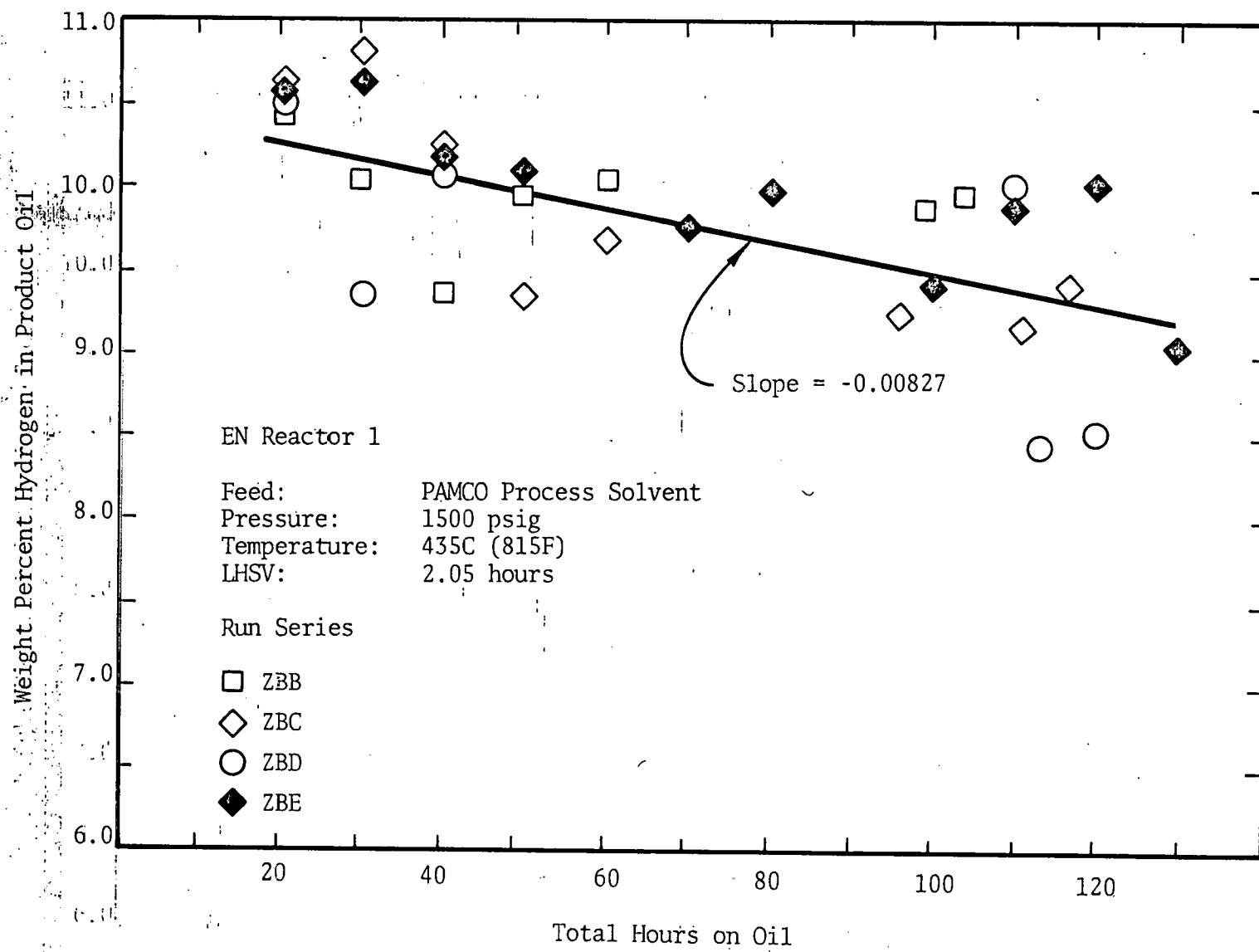


Figure 1. Hydrogenation Activity Response

TABLE I
 BOILING POINT DATA OF VARIOUS FEEDSTOCKS*
 (ASTM D-1160)

	Raw Anthracene Oil	20 wt% SRC-I in Anthracene Oil	Pamco Solvent Oil	30 wt% SRC-I in Pamco Solvent
I:B·P	123C(253F)	138C(280F)	159C(318F)	160C(320F)
10 Vol %	153 (306)	160 (320)	166 (331)	170 (338)
20	163 (325)	171 (340)	170 (338)	178 (352)
30	179 (354)	200 (392)	181 (358)	190 (374)
40	206 (403)	222 (432)	192 (378)	220 (428)
50	224 (435)	245 (473)	213 (415)	251 (484)
60	242 (468)	264 (507)	230 (446)	282 (540)
70	258 (496)	290 (554)	249 (480)	
80	276 (529)	340 (644)	265 (509)	
90	304 (579)			

*Determined at 50mm Hg vacuum

4. Scheppele, S. E., P. A. Benson, G. J. Greenwood, O. Grindstaff, T. Aczel and B. Bieber, A. C. S., Preprints, Division of Petroleum Chemistry, No. 4, Vol. 24, p. 963 (1979).
5. "Quarterly Report" for the period April 1 - June 30, 1980. DE-14876-3.

Task 2 - Catalyst Life Studies (H. J. Chang)

In the Catalyst Life Test Unit (CLTU), an air driven hydrogen compressor has been installed. This unit is able to compress low pressure hydrogen up to 10,000 psig. The purchase of expensive high pressure bottled hydrogen can be avoided in the future. The oil pump heads have been completely rebuilt and tested.

Five runs were made with Shell 324 NiMo/Al catalyst and two different feedstocks during this reporting quarter. Two runs, LTA and LTB, used a mixture of 40 wt% EDS (Exxon Donor Solvent process) vacuum gas oil/EDS raw solvent as feedstock. The other three runs, LTC, LTD and LTE used 30 wt% SRC/creosote oil as feedstock. The properties of the catalyst and feedstocks are shown in Tables II and III. Catalyst details were presented previously*.

Temperature variations in the longitudinal direction were negligible. However, more than 13F temperature differences between the center of the catalyst bed and the outer wall of the reactor were observed whenever the oils were first charged into the reactor at reaction temperature. These temperature differences were caused by exothermal reactions and were not observed in previous experimental runs. Apparently the high initial activity of the Shell 324 catalyst caused these increased temperature differences.

No decay in catalyst activity was observed when EDS process oil was used as feedstock. However, when 30 wt% SRC/creosote oil was used as feedstock, severe reactor plugging occurred after only four hours on oil at 371C(700F) or 399C(750F). Details follow.

Run Series LTA and LTB

These runs were designed to be part of an investigation of short-term catalyst decaying mechanisms, and the objectives of these runs were to generate results for use as the reference to determine the durations of the succeeding runs. The run conditions were as follows:

<u>Run Series</u>	<u>LTA</u>	<u>LTB</u>
Reactor	CLTU II	CLTU II
Catalyst	Shell 324 (Table I)	Shell 324 (Table I)
Feedstock	EDS process oil (Table II)	EDS process oil (Table II)
Reactor Outer Wall Temperature	399C(750F)	399C(750F), 426C(800F)
Center Catalyst Bed Temperature	410C(770F)	410C(770F), 437C(820F)

*"Quarterly Report" for the period January 1 - March 31, 1980, DE-14576-2.

TABLE II
CATALYST PROPERTIES

Catalyst Code	Shell-324
Manufacturer Chemical Composition	
NiO	2.7*
MoO ₃	13.2*
Physical Properties (1/16" Extrudate)	
Surface area m ² /gm	146(150*)
pore volume cc/gm	0.42(0.48*)
most frequent pore diameter(Å)	118
pore size distribution	
<u>Diameter (Å)</u>	<u>% pore volume in pore</u>
35-70	12
70-100	21
100-150	57
150-200	2
200-400	1
400-600	1
> 600	6
TOTAL	100

* Vendor's data

TABLE III
 PROPERTIES OF FEEDSTOCKS

	EDS Process ^a oil	30 wt% SRC/ creosote ^b
Density @ 70F, gm/ml	1.050	1.129
<u>Elemental composition, wt%</u>		
Carbon	88.21	90.70
Hydrogen	7.66	6.02
Nitrogen	0.72	1.25
Sulfur	0.70	0.54
Oxygen ^c	2.73	1.32
Ash	0.086	0.073
<u>Normal boiling point^d</u>		
Initial	211C(411F)	218C(424F)
5 Vol. %	224(435)	245(473)
10	231(448)	253(488)
20	245(473)	277(531)
30	283(541)	313(596)
40	321(610)	338(640)
50	388(731)	
60	451(843)	392(737)
61	454(850)	
70		436(816)
72		454(850)

a. Mixture of 40 wt% EDS vacuum gas oil/EDS raw solvent

b. Mixture of 30 wt% SRC-1/creosote oil

c. By difference

d. Determined from ASTM D 1160 at 20mm Hg vacuum.

Pressure	2000 psig	2000 psig
Liquid Volume Hourly Space Time (LVHST)	1.69, 2.63, 2.26 hrs.	2.26 hrs.
Hydrogen Flow	10,000 SCF/Bbl	10,000 SCF/Bbl
Run Duration	33.5 hrs.	261 hrs.

Run LTA was terminated after 33.5 hours on stream because of a faulty high pressure cylinder regulator. Run LTB was shut down on schedule at 261 hours on stream. The operation of Run LTB was essentially continuous, except that the gas and oil flows were off for one hour at 152 hours on oil to clean out a white solid material which plugged the gas exit line of the high pressure separation tank. The white crystal was found to be ammonium hydrogen sulfide (NH_4HS , melting point 20C) which solidified at cold ambient temperatures.

The results from sample analyses are listed in Tables IV and V. The hydrogen contents are 3.4 wt% higher in product oils than in the feedstock, and greater than 80 wt% nitrogen removal and 97 wt% sulfur removal were achieved at 399C(750F). At 426C(800F) operation, essentially all the nitrogen was removed. The weight percent nitrogen in product oil versus time on stream in Run LTB is plotted in Figure 2. The figure shows that the hydrodenitrogenation activity does not decay during the entire 261 hours on stream.

The feedstock used in these two experimental runs was easy to hydro-treat compared to other coal liquids which we have tested. Its rather high hydrogen-content (7.7 wt%) in part accounts for this observation. Because the catalyst demonstrated no activity decay over a 261 hour run, another feedstock was desirable.

Run Series LTC, LTD, and LTE

The feedstock in runs LTC, LTD and LTE, was changed to 30 wt% SRC/creosote oil. The objective of these later runs was to test this new oil as a potential feedstock for the future experimental runs of catalyst decaying mechanism studies.

The run conditions were as follows:

<u>Run Series</u>	<u>LTC</u>	<u>LTD</u>	<u>LTE</u>
Reactor	CLTU II	CLTU II	CLTU II
Catalyst	Shell 324 (Table I)	Shell 324 (Table II)	Shell 324 (Table III)
Feedstock	30 wt% SRC/creosote oil (Table III)		

TABLE IV

RESULTS FROM RUN LTA WITH SHELL 324 CATALYST
FEEDSTOCK EDS PROCESS OIL

Sample Number	Temp ^a (F)	Pressure (psig)	Space Time ^b (Volume Hrly)	Hydrogen (SCF/BBL)	Hours ^c on oil	wt%S ^d	wt%S ^e Removal	wt%N ^d	wt%N ^e Removal	wt%H ^d
Feed						0.70		0.72		7.66
LTA-1 ^f	750	2000	1.69	10,000	2					
LTA-2	750	2000	1.69	10,000	6	g		0.19	74	10.70
LTA-3	750	2000	2.63	10,000	12	g		0.12	83	11.56
LTA-4	750	2000	2.26	10,000	21	g		0.11	85	11.36
LTA-5	750	2000	2.26	10,000	29	g		0.11	85	11.13
LTA-6	750	2000	2.26	10,000	33.5	g		0.10	86	11.58

- a. This was the temperature at the outer wall of the reactor. The temperature at the center of the catalyst bed was 20F higher.
- b. This is a volume hourly space time (volume of catalyst/volume of oil per hour).
- c. Total hours which the catalyst has been contacted with oil at reaction temperature.
- d. Percent of sulfur, nitrogen or hydrogen in liquid product.
- e. % Removal = $100 \times (\text{fraction in feed} - \text{fraction in product}) / (\text{fraction in feed})$
- f. Not a representative sample
- g. Sulfur content lower than 0.02 wt%, which is the lower limit of our sulfur analyzer.

10. S. RESIST 10

TABLE V

RESULTS FROM RUN LTB WITH SHELL 324 CATALYST

FEEDSTOCK: EDS PROCESS OIL

Sample Number	Temp ^a (F)	Pressure (psig)	Space Time ^b (Volume Hrly)	Hydrogen (SCF/BBL)	Hours ^c on oil	wt%S ^d	wt%S ^e Removal	wt%N ^d	wt%N ^e Removal	wt%H ^d
Feed						0.70		0.72		7.66
LTB-1	750	2000	2.26	10,000	2	f		0.10	86	11.25
LTB-2	750	2000	2.26	10,000	6	f		0.13	82	11.32
LTB-3	750	2000	2.26	10,000	12	f		0.14	81	11.36
LTB-4	750	2000	2.26	10,000	20	f		0.14	81	11.23
LTB-5	750	2000	2.26	10,000	28	f		0.14	81	11.45
LTB-6	750	2000	2.26	10,000	36	f		0.11	85	11.52
LTB-7	750	2000	2.26	10,000	44	f		0.14	81	11.44
LTB-8	750	2000	2.26	10,000	56	f		0.12	83	11.06
LTB-9	750	2000	2.26	10,000	68	f		0.12	83	11.03
LTB-10	750	2000	2.26	10,000	80	f		0.13	82	11.19
LTB-11	750	2000	2.26	10,000	92	f		0.11	85	11.20
LTB-12	750	2000	2.26	10,000	104	f		0.11	85	11.03
LTB-13	750	2000	2.26	10,000	116	f		0.12	83	11.06
LTB-14	750	2000	2.26	10,000	128	f		0.12	83	11.07
LTB-15	750	2000	2.26	10,000	140	f		0.12	83	10.82
LTB-16	750	2000	2.26	10,000	152	f		0.12	83	11.08
LTB-17	750	2000	2.26	10,000	164	f		0.12	83	11.12
LTB-18	750	2000	2.26	10,000	177	f		0.11	85	11.21
LTB-19	750	2000	2.26	10,000	189	f		0.13	82	10.97
LTB-20	750	2000	2.26	10,000	201	f		0.10	86	11.18
LTB-21	750	2000	2.26	10,000	213	f		0.10	86	11.00
LTB-22	750 - 800	2000	2.26	10,000	224	f		0.03	96	11.35
LTB-23	800	2000	2.26	10,000	237	f		0.00	100	11.27
LTB-24	800	2000	2.26	10,000	248	f		0.00	100	11.26
LTB-25	800	2000	2.26	10,000	261	f		0.00	100	10.99

a. This was the temperature at the outer wall of the reactor. The temperature at the center of the catalyst bed was 20F higher.

b. This is a volume hourly space time (volume of catalyst/volume of oil per hour).

c. Total hours which the catalyst has been contacted with oil at reaction temperature.

d. Percent of sulfur, nitrogen or hydrogen in liquid product.

e. % Removal = $100 \times (\text{fraction in feed less fraction in product}) / (\text{fraction in feed})$

f. Not a representative sample

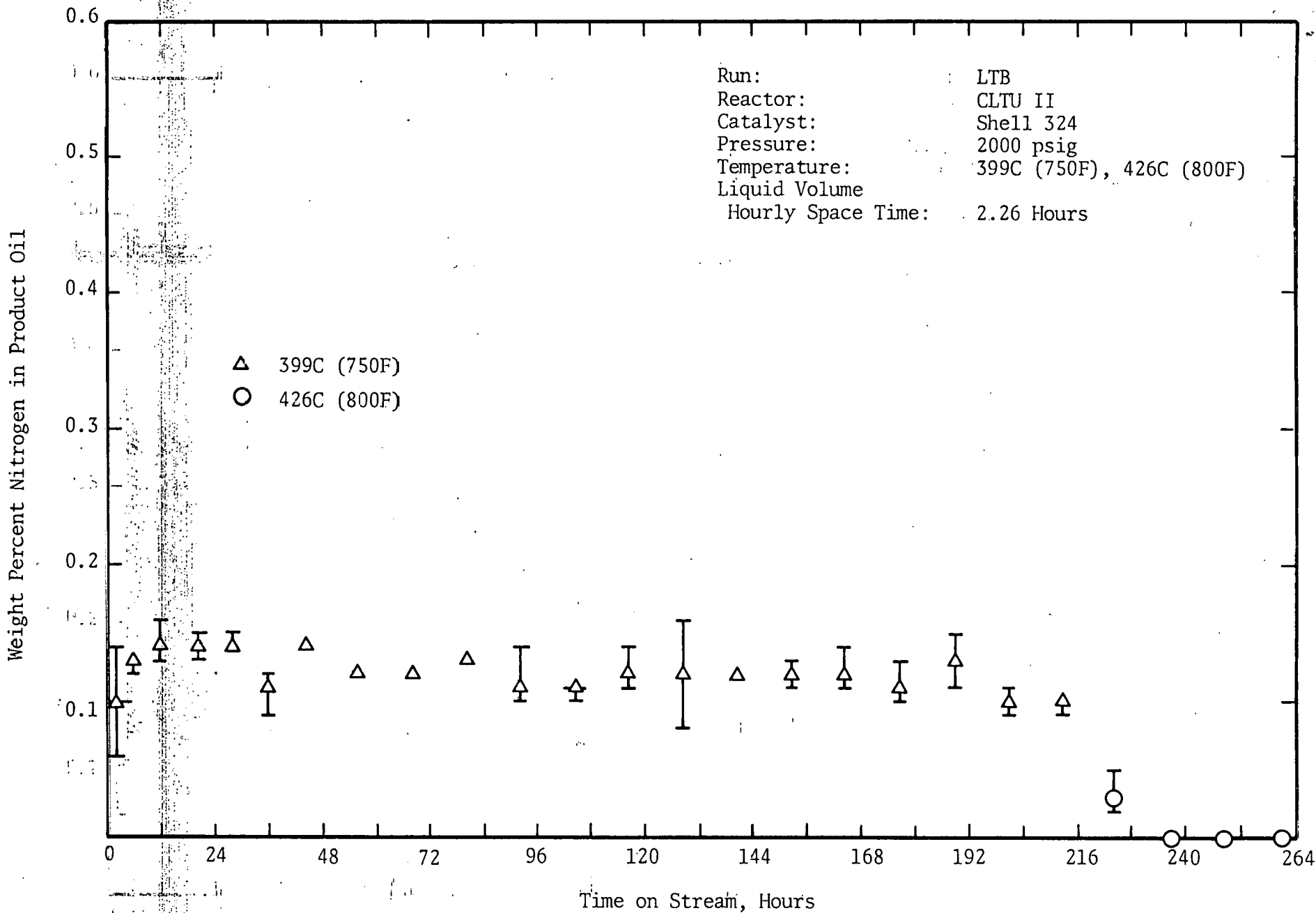


Figure 2. Hydrodenitrogenation Activity Response, Run LTB

Reactor Outer Wall Temperature	399C(750F)	399C(750F)	371C(700F)
Center Catalyst Bed Temperature	410C(770F)	410C(770F)	378C(713F)
Pressure	2000 psig	2000 psig	2000 psig
Liquid Volume Hourly Space Time	1.90 hours	1.90 hours	1.90 hours
Hydrogen Flow	10,000 SCF/bbl	10,000 SCF/bbl	10,000 SCF/bbl
Run Duration	25 hours	12 hours	12 hours

Unstable oil feed flow was suspected to be responsible for the reactor plugging which started at eight hours after start-up of Run LTC. The results from the analyses of Run LTC will not be available because of the unstable oil flow. Run LTD was designed to repeat Run LTC. Unfortunately, Run LTD also revealed plugging at four hours on stream. A lower temperature, 371C(700F), was used in Run LTE to avoid the plugging difficulties. Again, the reactor was plugged and shut down at 12 hours on stream.

The results from sample analyses of Runs LTD and LTE are shown in Table VI and VII, respectively. The catalyst had high hydrotreating power; it increased the hydrogen content of the feed by more than 3.5 wt%, and decreased the nitrogen content from 1.25 wt% in the feedstock to 0.30 wt% in the product oil for 399C(750F) operation. At 371C(700F), the activities were lower of course, but parallel to those at 399C(750F). The sulfur contents in the product oils at both temperatures were low. Catalyst activities decayed rapidly during the brief 12 hours operational periods. This was caused by the rapid coking reactions probably caused by the silica content of the catalyst.

The temperature difference in the radial direction for the experimental run at 371C(700F) was only 7C(13F), where as the difference at 399C(750F) operation was 11C(20F). This was consistent with the fact that the reactivities were greater at the higher temperature.

Other studies using SRC-I with creosote oil have demonstrated the difficulty in hydrotreating this feedstock.* In fact, prehydrotreatment of only the creosote solvent before mixing with SRC-I was used to prepare a feedstock that could be processed.

Conclusions

- 1) A high pressure hydrogen compressor has been installed and operated for economical reasons.
- 2) Five experimental runs were made during this reporting quarter. None of the results from these runs met the original objectives. However, these have generated valuable results for the future studies of coking mechanisms.
- 3) Pressure, temperature, liquid and gas flow rates have been very stable during these runs.

* "Commercial Scale Expanded Bed Hydroprocessing of Solvent Refined Coal Extract", Interim Technical Progress Report, November, 1979, FE-2038-17.

TABLE VI

RESULTS FROM RUN LTE WITH SHELL 324 CATALYST

FEEDSTOCK: 30 wt% SRC/CREOSOTE

Sample Number	Temp ^a (F)	Pressure (psig)	Space Time ^b (Volume Hrly)	Hydrogen (SCF/BBL)	Hours ^c on oil	wt%S ^d	wt%S ^e Removal	wt%N ^d	wt%N ^e Removal	wt%H ^d
Feed						0.54		1.25		6.02
LTE-1 ^f	700	2000	1.90	10,000	2					
LTE-2	700	2000	1.90	10,000	6	0.06	89	0.49	61	8.91
LTE-3	700	2000	1.90	10,000	12	0.08	85	0.60	52	8.71

- a. This was the temperature at the outer wall of the reactor. The temperature at the center of the catalyst bed was 13F higher.
- b. This is a volume hourly space time (volume of catalyst/volume of oil per hour).
- c. Total hours which the catalyst has been contacted with oil at reaction temperature
- d. Percent of sulfur, nitrogen or hydrogen in liquid product.
- e. % removal = $100 \times (\text{fraction in feed less fraction in product}) / (\text{fraction in feed})$
- f. Not a representative sample.

TABLE VII

RESULTS FROM RUN LTD WITH SHELL 324 CATALYST

FEEDSTOCK: 30 wt% SRC/CREOSOTE OIL

Sample Number	Temp ^a (F)	Pressure (psig)	Space Time ^b (Volume Hrly)	Hydrogen (SCF/BBL)	Hours ^c on oil	wt%S ^d	wt%S ^e Removal	wt%N ^d	wt%N ^e Removal	wt%H ^d
Feed						0.54		1.25		6.02
LTD-1 ^f	750	2000	1.90	10,000	2					
LTD-2	750	2000	1.90	10,000	6	0.02	96	0.18	86	9.87
LTD-3	750	2000	1.90	10,000	12	0.04	93	0.30	76	9.67

- a. This was the temperature at the outer wall of the reactor. The temperature at the center of the catalyst bed was 20F higher.
- b. This is a volume hourly space time (volume of catalyst/volume of oil per hour).
- c. Total hours which the catalyst has been contacted with oil at reaction temperature.
- d. Percent of sulfur, nitrogen or hydrogen in liquid product.
- e. % Removal = $100 \times (\text{fraction in feed less fraction in product}) / (\text{fraction in feed})$
- f. Not a representative sample

- 4) Significant temperature differences between the catalyst bed center and the reactor outer wall have been observed for the first time. The high initial activity of the catalyst resulted in these significant temperature differentials.
- 5) No deactivation in catalyst activities was observed when EDS process oil was used as feedstock. However, when the SRC/creosote oil was used as feedstock, rapid coking reactions resulted in severe reactor plugging in only a few hours on stream.
- 6) The results show that the Shell 324 catalyst demonstrates bifunctional activities when hydrotreating the hydrogen deficient SRC/creosote mixture. This results in relatively high hydrogen content in product oils, and yet rapid coke formation in the catalyst bed.
- 7) These results suggest that in order to meet the objectives of investigating the short-term catalyst decaying mechanisms, a solvent more compatible than creosote oil has to be used in the future studies.

Work Forecast

During the coming quarter, one or more experimental runs of 300 hours and less will be made using Shell 324 NiMo/Al catalyst and 30 wt% SRC/PAMCO process solvent as feedstock. PAMCO process solvent is available and derived from a coal liquefaction process. It is expected to be a compatible solvent in hydrotreating SRC. These runs will be part of a series investigation of short-term catalyst decaying mechanisms, and the results will be used as the reference to determine the durations of the succeeding runs.

Task 2-c Catalyst Regeneration (A. N. Tayrien)

The catalyst used in experimental run CDS was regenerated by controlled oxidation at 550C to remove coke deposits. This oxidation process occurred after lengthy Soxhlet extraction to remove organic material. A subsequent experimental run (CRS) was conducted in Reactor EN-1 to assess the hydrotreating activity after regeneration. The conditions were essentially the same as those that were utilized in experimental run CDS: 427C(800F), 1500 psig nominal pressure, 2.75 hours space time (liquid volume hourly basis). The run duration was 101 hours of continuous liquid-catalyst contact with a scheduled shutdown. The catalyst was regenerated HDN-30 (Ni-Mo-Al₂O₃).* The feedstock consisted of a ten volume percent Synthoil II liquid with 90 volume percent raw anthracene oil. This feedstock was chosen specifically to emphasize catalyst deactivation.

Partial analyses of the feedstock and product oils have been completed, but full results will be presented when the experimental series is finished. Catalyst samples are being retained from each experimental run, both before and after regeneration to assess the property changes as a function of history and treatment.

*Catalyst properties found in Quarterly Report for period April 1 - June 30, 1980, DE-14876-3.

RECEIVED BY TIC FEB 9 1981