

CATALYTIC HYDROGENATION OF COAL-DERIVED LIQUIDS

Interim Report for the  
Period June, 1977 - August, 1977

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## OBJECTIVE

It is the object of this research to convert coal to clean distillate fuels. This program will be limited to research on the product from existing coal liquefaction processes. Liquefied coal will be converted by a catalytic hydrogenation at elevated temperatures and pressures. Samples of the products from PAMCO, H-Oil, COED and SYNTHOIL will be obtained. They will be heated and pumped, with and without solvent, into a catalytic reactor in the presence of hydrogen and other reducing gases. Variables to be investigated will include temperature, pressure, space velocity, hydrogen-to-oil ratio, chemical nature of the solvent. The catalysts to be studied will include nickel molybdate and sulfide, nickel tungstate and other combinations on carriers such as mordenites and other molecular sieve types.

## ABSTRACT

For COED pyrolysis oil, the screening of available commercial catalysts was completed. Cobalt molybdates are the best for desulfurization, nickel molybdates the best for denitrogenation. For PAMCO Solvent Refined Coal (SRC) Vacuum Flash Feed, the best desulfurization to date has been obtained with cobalt molybdate catalysts. Analyses including desulfurization, denitrogenation and ASTM distillations for nine previously reported runs and eleven new runs are reported. For SYNTHOIL, four more runs were completed and correlated with the previously reported forty-five runs. The evaluation of two more commercial catalysts and two MSU-fabricated catalysts was completed.

## NUGGET

After having tested the available commercial catalysts recommended for hydrotreating on Solvent Refined Coal (SRC), we formulated a catalyst which exceeded any of the commercial catalysts for combined desulfurization - denitrogenation. MSU-STK-5-2-2-E-1/16" containing 0.26% NiO, 1.15% CoO and 13.1% MoO<sub>3</sub> on Ketjen -003 alumina gave 55% denitrogenation (1.14% to 0.51% N<sub>2</sub>), 65% desulfurization (0.72% to 0.25% S). The best commercial catalyst for SRC was Harshaw HT-400-E-1/16" which contained 3% CoO, 15% MoO<sub>3</sub> on alumina and gave 34% denitrogenation and 54% desulfurization.

COED PYROLYSIS OIL - S. Kujawa

SUMMARY OF TECHNICAL PROGRESS

Previously on this research it had been reported that the Harshaw Catalyst, Ni4401, consisting of 6% NiO, 19% WO<sub>3</sub>, 25% Al<sub>2</sub>O<sub>3</sub>, and 50% SiO<sub>2</sub>, in a presulfided 1/12" extrusion, gave the best overall catalyst performance in terms of hydrotreating and hydrocracking COED Pyrolysis Oil. This conclusion was reached on the basis of batch and continuous runs at 450°C, and high hydrogen pressure. However, in the period since the above runs were made, more recent runs indicated that the severity of the screening runs should be reduced to obtain a better idea of the intrinsic activity of the catalysts. The reduced temperature runs were made at an approximate average catalyst bed temperature of 415°C, and a hydrogen feed rate of 10,000 scf/bbl at 800 psig.

Twelve continuous runs were made during this quarter, which along with the seven made the previous quarter, complete the screening runs of commercial catalysts for this project. Six cobalt molybdate catalysts were tested along with five nickel tungsten catalysts, and five nickel molybdate catalysts.

Sulfur removal was best effected with cobalt molybdate catalysts. At a space velocity of one and a half, Harshaw HT 400, Shell 344, and Cyanamid HDS20A each removed over 90% of the sulfur in the feed. The Harshaw and Shell catalysts performed slightly better than the Cyanamid catalyst.

The nickel molybdate catalysts were the best for nitrogen removal. Cyanamid HDS9A gave the best apparent nitrogen removal with Harshaw HT500 closely following. The Cobalt moly catalysts did fairly good nitrogen removal also, with Harshaw HT 400 and Shell 344 giving results similar to Harshaw HT 500 at the lower space velocities.

The nickel tungsten catalysts tested were poor compared with Cobalt moly

and nickel moly catalysts for heteroatom removal. For nitrogen removal, both the nickel tungsten and cobalt moly catalysts showed a dependence on average pore size. This was not apparent with nickel moly catalysts.

In general, the ASTM D-86 distillation curves gave a liquid product distribution that was similiar for each catalyst. The cobalt molybdate catalysts consistently gave slightly more volume percent boiling less than 700<sup>0</sup>F, the majority of this increase being in the boiling range of 600-700<sup>0</sup>F. Several of the nickel tungsten catalysts showed a slight tendency to produce more liquid in the up to 425<sup>0</sup>F boiling range, this seemingly due to cracking of the 600-700<sup>0</sup>F. range as evident from the lower volume percent in this range along with lower overall distillate yield. The nickel molybdate catalysts yielded large amounts of product in the range 425-600<sup>0</sup>F with low amounts in the up to 425<sup>0</sup>F range. A possible conclusion from this is that the nickel moly catalysts are making less gas, which would be very desirable.

#### OBJECTIVES DURING THIS QUARTER

During the period June through August, 1977, the following was accomplished:

1. Continuous screening runs were completed on the commercial catalysts:  
Nickel Molybdates: Shell 324, Harshaw HT-100 and HT-500, Houdry HR-811, and Cyanamid HDS9A.  
Nickel Tungstates: Harshaw 4401, 4301, and 4303; Ketjen HC-5 and Ketjen-fine 330-3E.
2. Analyses were competed on the products of the above runs and of those using the cobalt molybdate catalysts: Harshaw 0401, and HT-400; Shell 344, Cyanamid HDS-20A; Houdry HR-801, and CCI C20-6.

## EXPERIMENTAL PROCEDURE

The experimental runs were made with a continuous reactor made from a 30" long piece of 1" Inconel schedule 80 pipe. The pressure was approximately 800 psig and the hydrogen feed rate was approximately 10,000 scf/bbl (1781 NL/L). The catalyst bed consisted of 80 ml. of catalyst mixed with 80 ml of inert Denstone support and was preceded in the reactor by inert support used as a preheating section. During the runs it was attempted to keep the top and bottom of the bed at 410°C; usually the middle of the bed temperature was such that the average of the three was approximately 415°C. Two to three reactor volumes of feed were run through the reactor between changes of variables to allow the reactor operation to steady out, usually three samples were taken during a run with the total amount of feed being between 1200 and 1700 ml. No attempt was made to check the initial activity decay of the catalyst.

## RESULTS AND DISCUSSION

### Cobalt Molybdate Catalysts

The experimental results using cobalt moly catalysts are presented in Figures 1-1 and 1-2, and Tables 1-I and 1-IV. Harshaw HT 400 and Shell 344 gave the best sulfur removal, followed closely by Cyanamid HDS20A. At the lower space velocities, nitrogen removal was as good for these catalysts as the nickel molys shown later in the report.

In Figure 1-2, the nitrogen removal for the above three catalysts increases with the calculated average pore diameters. This suggests that nitrogen removal is severely limited by pore diffusion.

Table 1-I gives the distribution of liquid product according to boiling ranges. Shell 344 and Harshaw HT400 appear to give the highest amount of low

boiling products, however, the boiling curves did not vary much in any of the products from the six catalysts tested.

#### Nickel Tungsten Catalysts

All of the nickel tungsten catalysts had approximately 6% Ni and 19% W on them. Harshaw 4401, 4301, and Ketjenfine 330-3E were silica alumina supports, which were intended to increase hydrocracking activity. The silicated supports were characterized by a much smaller average pore size than the non-silicated catalysts.

Sulfur removal for these catalysts was lower than the cobalt molybdate catalysts. Also, nitrogen removal was less; in many cases there being more nitrogen in the product than in the feed. The characteristics of heteroatom removal exactly followed pore size for these catalysts, the large pore non-silicated catalysts, Ketjen HC-5 and Harshaw 4303, doing the best job.

In Figure 1-3, it appears that the slope for Ketjen HC-5 is negative, contrary to the trend of the rest of the catalysts. Although this could be due to effects of poisoning or film diffusion, what is more likely to have happened is the samples at the lower and higher space velocities were switched during labeling, thus giving the pictured results.

From Table 1-II, it appears that the silicated supports yielded a higher percent of lighter products in certain boiling ranges. Harshaw 4401 appeared to give a higher volume in the 600-700<sup>o</sup>F range, and Ketjenfine 330-3E gave a larger volume of IBP to 425<sup>o</sup>F products.

A special note about Harshaw 4401. Previously, in these reports, it had been thought that this catalyst gave the best overall results. However, the runs this was based on occurred at 450<sup>o</sup>C. with an un-diluted catalyst bed of 100 ml volume. When it was tested at the less severe conditions of this series

of runs, it was found to be much less active towards heteroatom removal than the newer generation of catalysts. This example shows the advisability of testing initially at less severe temperatures to obtain a better idea of the catalyst's intrinsic activity.

#### Nickel Molybdate Catalysts

Figures 1-5 and 1-6 and Tables 1-III and 1-VI show the results of the continuous tests for the five nickel moly catalysts. Sulfur removal and liquid product yield was poorer than the cobalt moly catalysts. Nitrogen removal was the best so far in a continuous run. Cyanamid HDS9A took out the most nitrogen, closely paralleled by Harshaw HT 500 and Houdry HR 811; however, the Houdry catalyst appeared to be poisoned quickly during the course of the run. Interestingly, Shell 324 removed the most sulfur of the nickel moly catalysts while being the second worst nitrogen remover. No reason for this is apparent though. The Shell 324 catalyst did give the best liquid product yields, but the Cyanamid HDS9A catalyst gave a higher percent in the desirable 425-600<sup>0</sup>F boiling range than any other catalyst.

#### OVERALL PERFORMANCE AND CONCLUSIONS

From the experimental evidence, it appears that the best nickel moly and cobalt moly catalysts show similar activity for hydrotreating COED Pyrolysis oil; the cobalt molybdate catalysts removing more sulfur, while the nickel moly catalysts show more activity for nitrogen removal. The nickel tungsten catalysts show lower activity than the cobalt and nickel moly catalysts for hydrotreating.

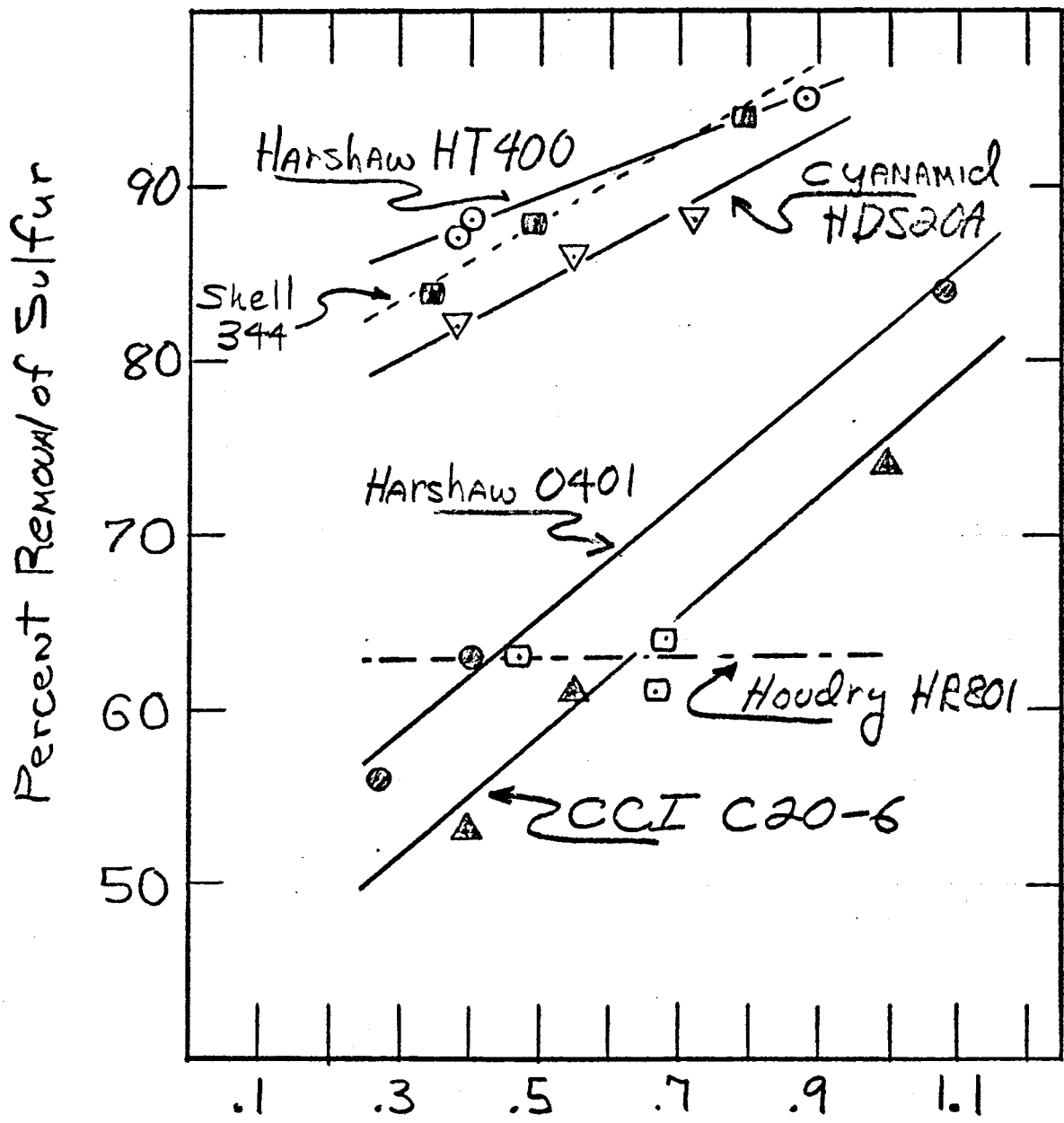
From the limited average pore size data available for the catalysts tested, it appears that nitrogen removal, and to a lesser extent sulfur removal, is very limited by pore diffusion. To obtain a catalyst that will remove

nitrogen to the low levels necessary for further processing a study of the effects of pore size distribution will have to be made to obtain data on catalyst design.

A possible processing method would be to run the feed over a cobalt moly catalyst to remove sulfur, and then over a nickel moly catalyst to remove nitrogen. This method might bring the heteraton levels down to an acceptable point without severely increasing the processing conditions.

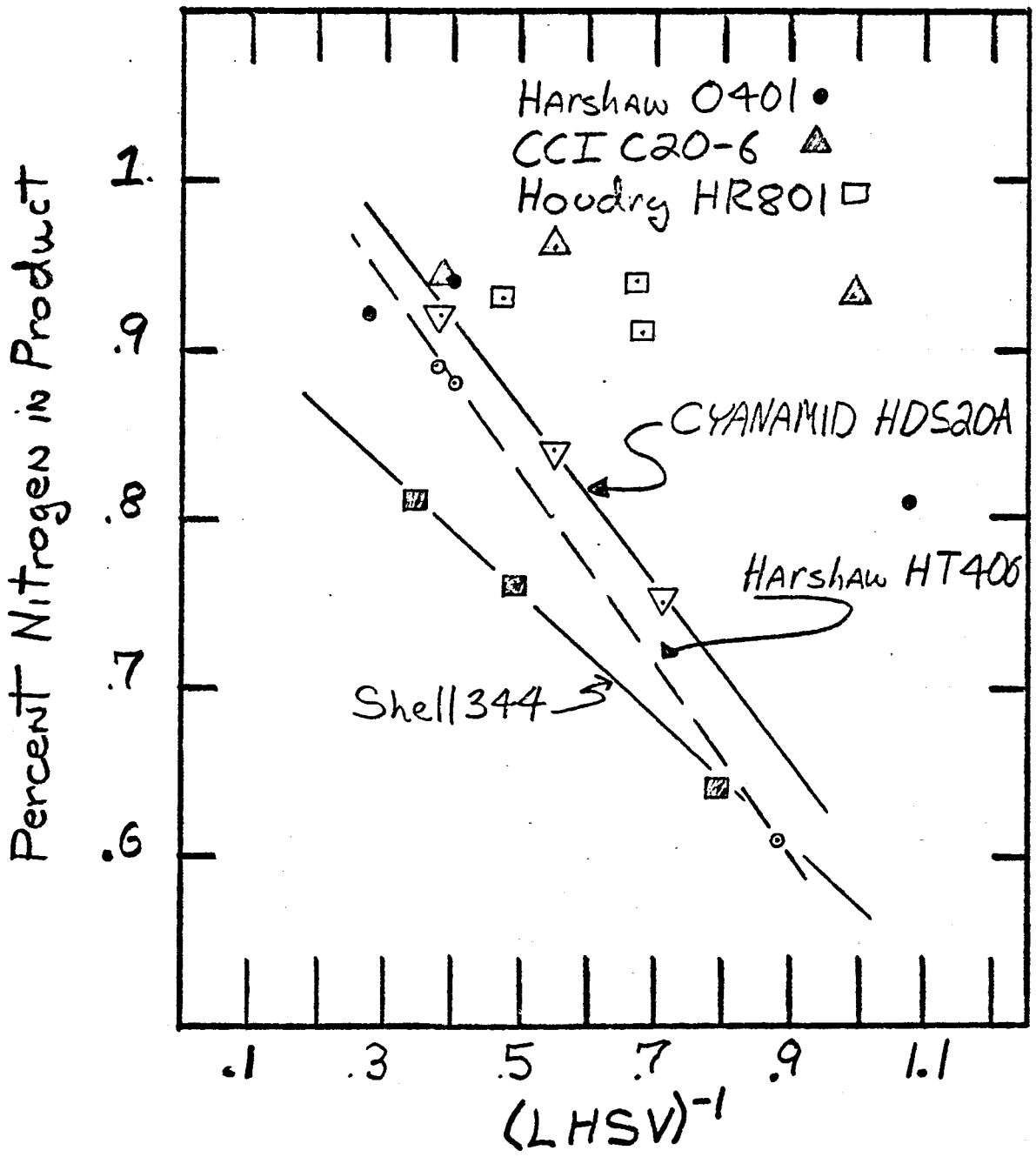
#### PROJECT FORECAST - COED PYROLYSIS OIL

During the next quarter, six catalysts will be tested consisting of nickel, cobalt and molybdenum impregnated on six different supports. The data from these tests will be used to determine what effects the support has on the reaction. Five nickel-cobalt-tungsten catalysts on a Ketjen support will be tested to see if they are as active as the nickel cobalt molybdenum catalysts.



(LHSV)<sup>-1</sup>  
Commercial Cobalt Molybdate Catalysts

Figure I-I

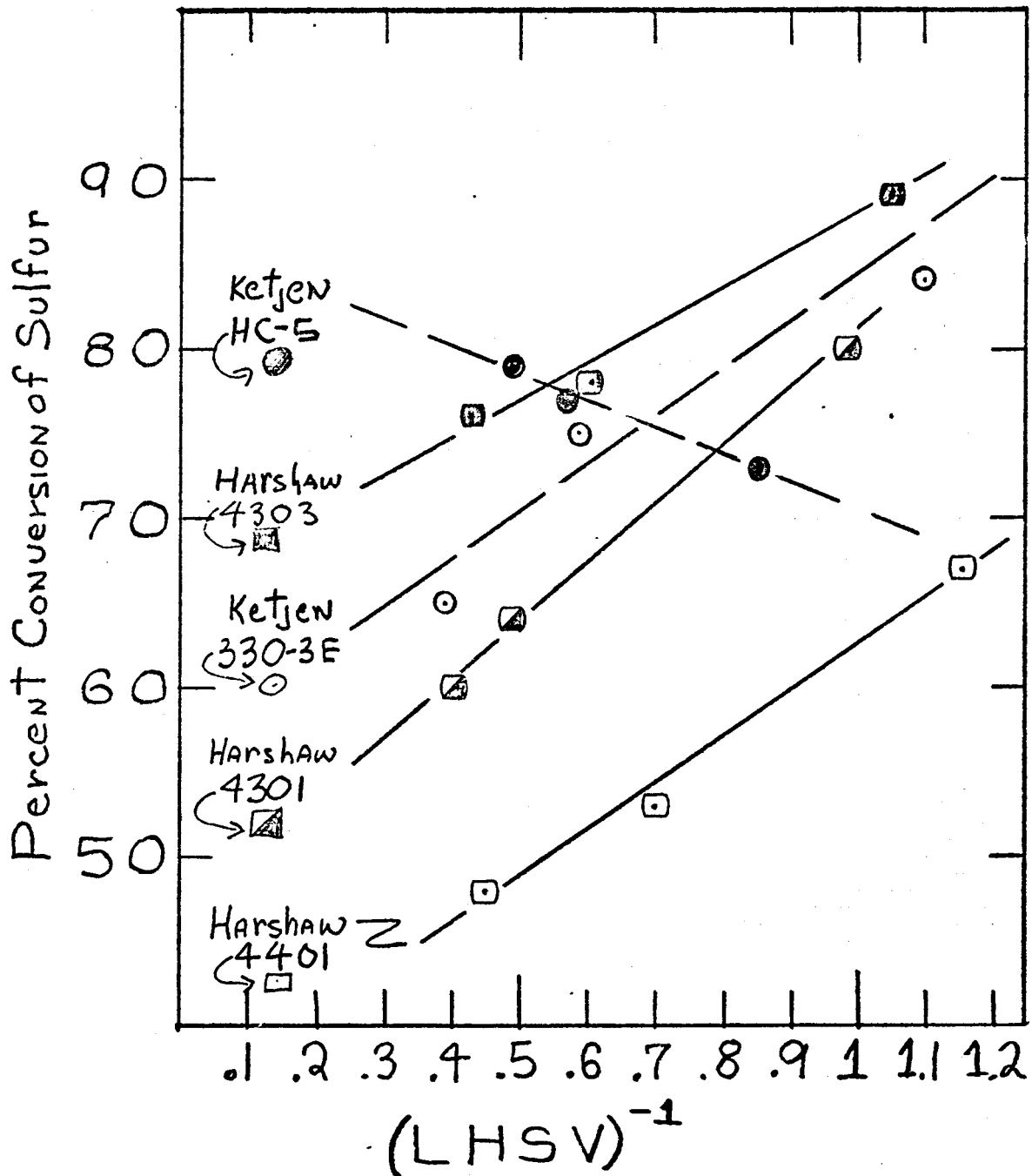


Commercial Cobalt Molybdate Catalysts  
Figure 1-2

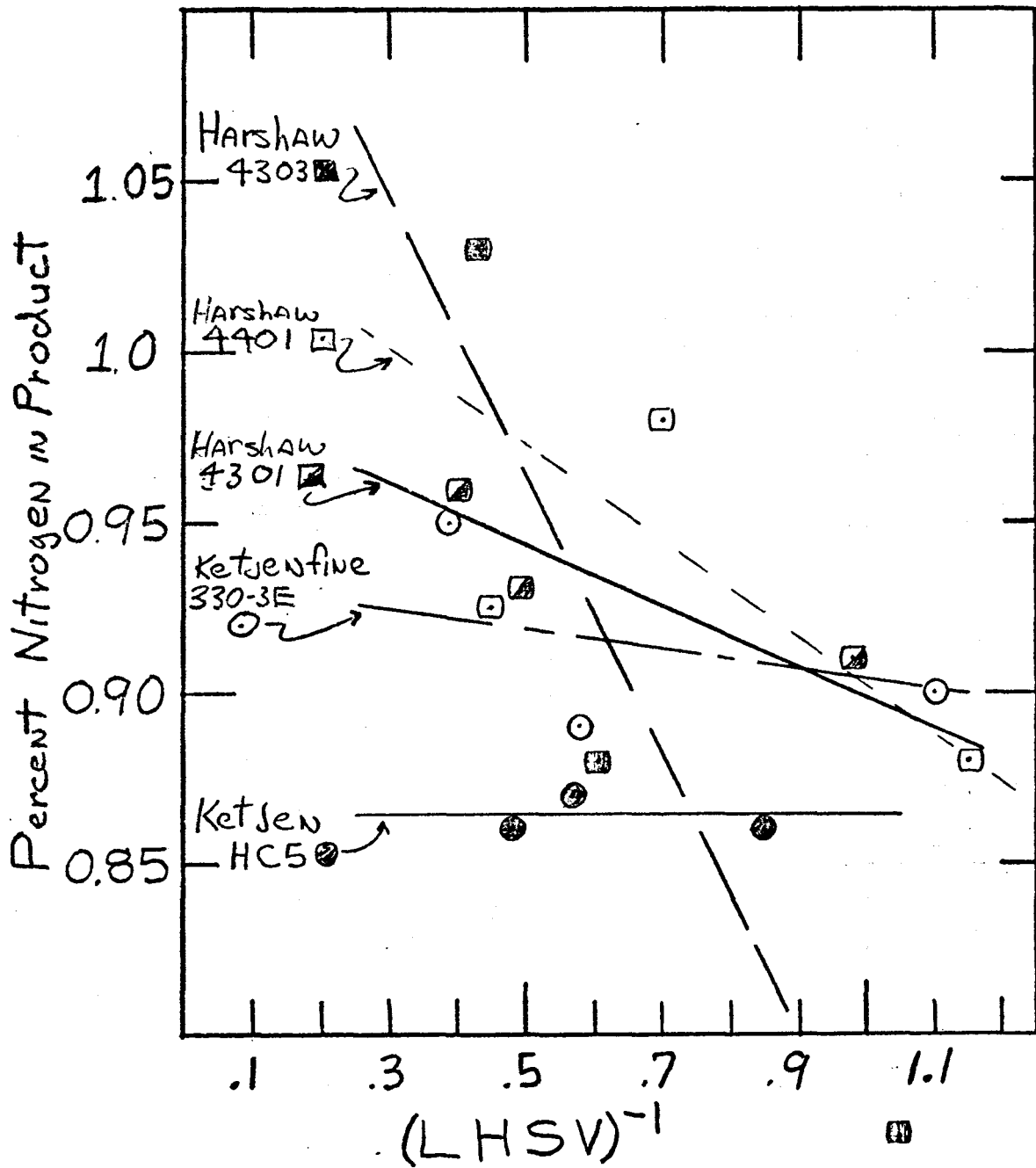
TABLE 1-I

LIQUID PRODUCT DISTRIBUTION FROM ASTM DISTILLATIONCobalt Moly Catalysts

CATALYST	LHSV	IBP-425	425-600	600-700	REC.
Harshaw 0401	.93	19	32	20	74
	2.5	16	23	31	74
	3.72	14	27	28	72
Harshaw HT400	1.13	-			-
	2.46	12	32	30	76
	2.64	-			-
Shell 344	1.27	-	-	-	-
	2.06	19	28	29	78
	2.84	16	27	25	70
Houdry HR801	1.46	24	23	20	73
	1.49	18	22	25	71
	2.13	18	24	18	72
CCI-C20-6	1.01	20	24	24	73
	1.82	17	21	22	66
	2.58	16	21	29	70
Cyanamid HDS20A	1.38	18	27	21	69
	1.81	16	27	20	67
	2.66	17	25	22	69



Commercial Nickel Tungsten Catalysts.  
Figure 1-3

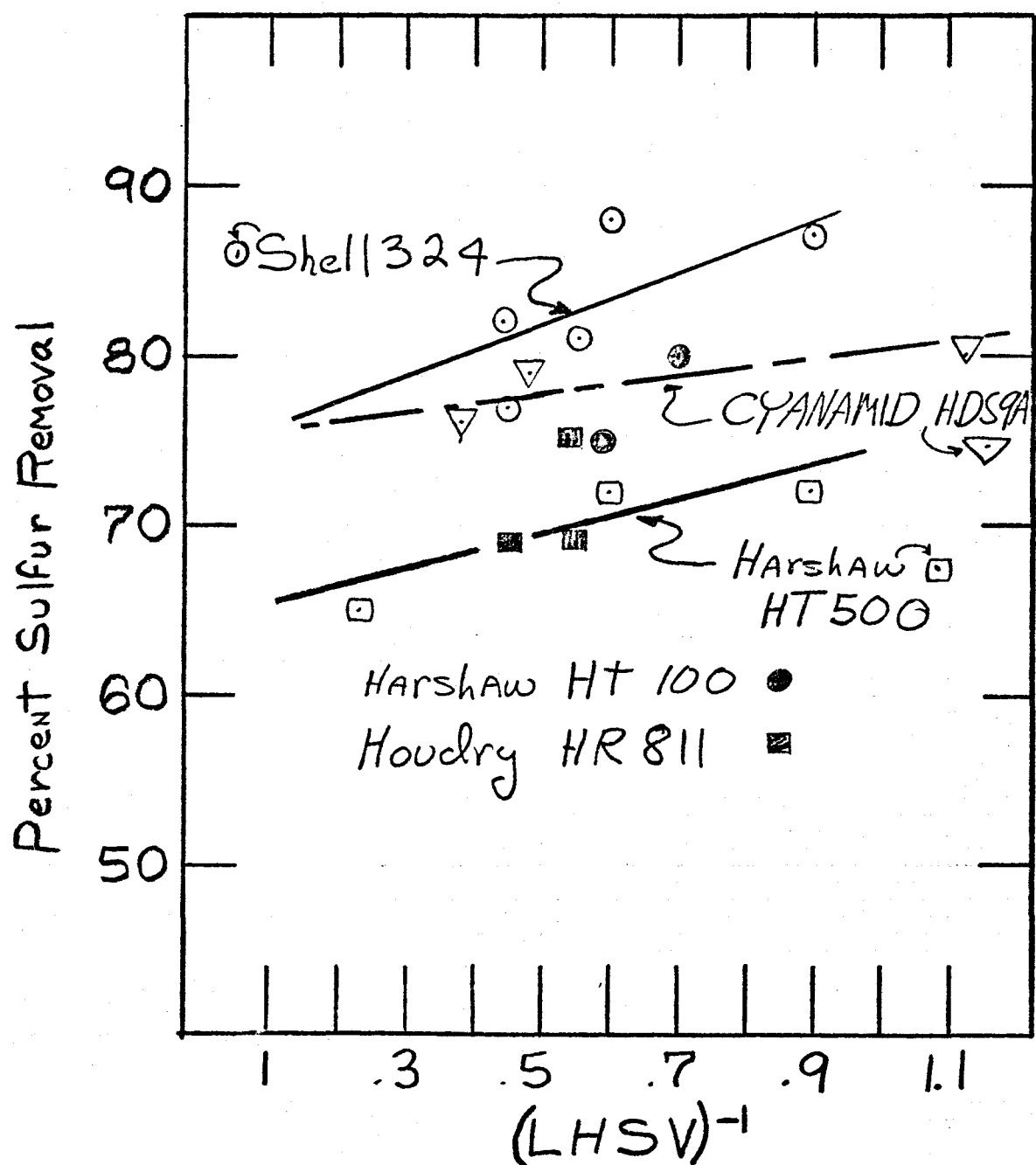


Commercial Nickel Tungsten Catalysts  
Figure 1-4

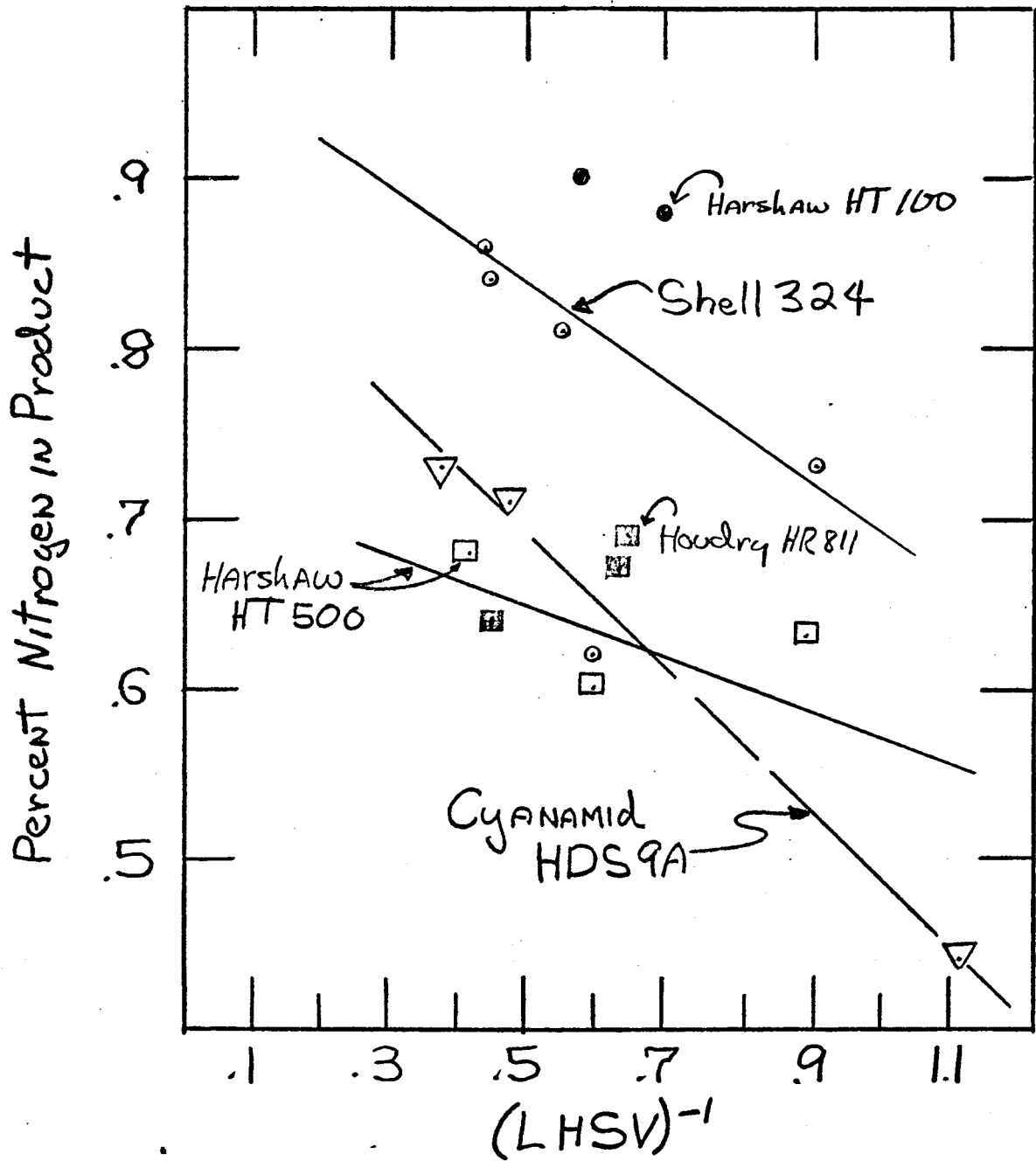
TABLE 1-II

LIQUID PRODUCT DISTRIBUTION FROM ASTM DISTILLATIONNickel Tungsten Catalysts

CATALYST	LHSV	IBP-425	425-600	600-700	REC.
Harshaw 4401	.86	19	26	24	74
	1.43	8	22	30	63
	2.23	10	21	29	66
Harshaw 4301	1.02	12	26	16	61
	2.06	9	24	21	60
	2.46	5	29	24	64
Harshaw 4303	.95	17	29	17	68
	1.65	16	25	17	64
	2.35	13	27	16	62
Ketjenfine 330-3E	.91	25	22	16	68
	1.71	22	23	22	72
	2.56	8	25	24	63
Ketjen HC-5	1.17	20	22	23	70
	1.75	22	20	19	66
	2.09	10	33	19	66



Commercial Nickel Molybdate Catalysts  
Figure 1-5



Commercial Nickel Molybdate Catalysts  
Figure 1-6

TABLE 1-III  
LIQUID PRODUCT DISTRIBUTION FROM ASTM DISTILLATION

Nickel Molybdate Catalysts

CATALYSTS	LHSV	IBP-425	425-600	600-700	REC.
Shell 324	1.63	22	28	19	73
	1.77	19	25	21	68
	1.1	20	29	17	69
	2.24	13	28	19	64
	2.26	14	28	20	66
Harshaw HT100	1.41	13	28	17	63
	1.71	10	27	20	62
Harshaw HT500	1.12	11	27	23	68
	1.66	11	21	28	64
	2.31	7	28	17	60
Cyanamid HDS9A	.89	11	35	28	80
	2.08	11	30	18	65
	2.63	7	29	16	60
Houdry HR811	1.54	14	24	17	63
	1.56	15	23	20	68
	2.17	11	28	17	64

TABLE 1-IV

## COBALT MOLYBDATE CATALYST PERFORMANCE

CATALYST AVG. BED TEMP. (°C) H <sub>2</sub> FEED RATE (NL/L)	LIQUID HOURLY SPACE VELOCITY	% S IN PRODUCT	% REMOVAL OF SULFUR	% N IN PRODUCT	% REMOVAL OF NITROGEN	ASTMD-86 DISTILLATION OF LIQUID PRODUCT VOL. % BOILING BELOW			
						425F	600F	700F	RECOVERY
Harshaw 0401-T-1/8"	.93	.36	84	.81	10	19	51	71	74
420	2.5	.86	63	.94	0	16	39	70	74
1761	3.72	1.02	56	.92	0	14	41	69	72
Harshaw HT-400-E-1/16"	1.13	.12	95	.61	32	-	-	-	-
414	2.46	.27	88	.88	2	12	44	74	76
1700	2.64	.29	87	.89	1	-	-	-	-
Shell 344-E-1/16"	1.27	.13	94	.64	29	-	-	-	-
408	2.06	.27	88	.76	16	19	47	76	78
1616	2.84	.36	84	.81	10	16	43	68	70
Houdry HR-801-E-1/16"	1.46	.83	64	.91	0	24	47	67	73
396	1.49	.89	61	.94	0	18	40	65	71
1722	2.13	.86	63	.93	0	18	42	60	72
CCI-C20-6-E-1/16"	1.01	.59	74	.93	0	20	44	68	73
404	1.82	.89	61	.96	0	17	38	60	66
1769	2.58	1.08	53	.94	0	16	37	66	70
Cyanamid-HDS20A	1.38	.28	88	.75	18	18	45	66	69
413	1.81	.31	86	.84	9	16	43	63	67
1840	2.66	.40	82	.92	0	17	42	64	69

CATALYST DESCRIPTIONS	% Co	% Mo	SUPPORT	S.A.	P.V.	AVG. PORE SIZE
Harshaw 0401	3	9	Silica Alumina	160	.4	100 Å
Harshaw HT400	3	15	Alumina	220	.5	91 Å
Shell 344	2.4	9.9	Alumina	195	.6	123
Houdry HR-801	3	15	Alumina	300	-	-
CCI C20-6	3	15	Alumina	-	-	-
Cyanamid HDS20A	5	16.2	Alumina	230	.52	90 Å

TABLE 1-V

NICKEL TUNGSTEN CATALYST PERFORMANCE

CATALYST AVG. BED TEMP. (°C) H <sub>2</sub> FEED RATE (NL/L)	LIQUID HOURLY SPACE VELOCITY	% S IN PRODUCT	% REMOVAL OF SULFUR	% N IN PRODUCT	% REMOVAL OF NITROGEN	ASTMD-86 DISTILLATION OF LIQUID PRODUCT VOL. % BOILING BELOW			
						425F	600F	700F	RECOVERY
Harshaw 4401	.86	.76	67	.885	2	19	45	69	74
415	1.43	1.08	53	.98	0	8	30	60	63
1844	2.23	1.2	48	.925	0	10	31	60	66
Harshaw 4301	1.02	.47	80	.91	2	12	38	54	61
413	2.06	.81	64	.93	0	9	33	54	60
1757	2.46	.92	60	.96	0	5	34	58	64
Harshaw 4303	.95	.26	89	.77	16	17	46	63	68
414	1.65	.5	78	.88	4	16	41	58	64
1779	2.35	.54	76	1.03	0	13	40	56	62
Ketjen 330-3-E	.91	.57	75	.90	2	25	47	63	68
412	1.71	.57	75	.89	3	22	45	67	72
1746	2.56	.80	65	.95	0	8	33	57	63
Ketjen HC-5	1.17	.61	73	.86	7	20	42	65	70
414	1.75	.52	77	.87	5	22	42	61	66
1798	2.09	.48	79	.86	7	10	43	62	66

<u>CATALYST DESCRIPTIONS</u>	<u>% N.</u>	<u>% W.</u>	<u>SUPPORT</u>	<u>S.A.</u>	<u>P.V.</u>	<u>AVG. PORE SIZE</u>
Harshaw 4401	6	19	50% SiO <sub>2</sub> , 25% Al <sub>2</sub> O <sub>3</sub>	-	-	-
Harshaw 4301	6	19	Silica Alumina	228	.37	65 A
Harshaw 4303	6	19	Alumina	152	.54	142 A
Ketjen 330-3E	6.6	19.8	Silicia Alumina	193	.43	89 A
Ketjen HC-5	6.5	21	Alumina	200	.62	124 A

TABLE 1-V I

NICKEL MOLYBDATE CATALYST PERFORMANCE

CATALYST AVG. BED TEMP. (°C) H <sub>2</sub> FEED RATE (NL/L)	LIQUID HOURLY SPACE VELOCITY	% S IN PRODUCT	% REMOVAL OF SULFUR	% N IN PRODUCT	% REMOVAL OF NITROGEN	ASTMD-86 DISTILLATION OF LIQUID PRODUCT VOL. % BOILING BELOW			
						425F	600F	700F	RECOVERY
Shell 324-E-1/16" 414 (80 CC bed) 1704	1.63	.27	88	.62	33	22	50	69	73
	1.77	.44	81	.81	12	19	44	65	68
Shell 324-E-1/16" 412 (68 CC bed) 1765	1.1	.29	87	.73	21	20	49	66	69
	2.24	.53	77	.84	9	13	41	60	64
	2.26	.41	82	.86	7	14	42	62	66
Harshaw HT-100-E-1/12" 414 1831	1.41	.46	80	.88	4	13	41	58	63
	1.71	.56	75	.90	2	10	37	57	62
Harshaw HT-500-E-1/8" 412 1768	1.12	.64	72	.63	32	11	38	61	68
	1.66	.63	72	.6	35	11	32	60	64
	2.31	.80	65	.68	26	7	35	52	60
Cyanamid HDS-9A 414 1812	.89	.45	80	.44	52	11	46	74	80
	2.08	.48	79	.71	23	11	41	59	65
	2.63	.54	76	.73	21	7	36	52	60
Houdry HR811-E-1/8" 414 1856	1.54	.70	69	.69	25	14	38	55	63
	1.56	.58	75	.67	27	15	38	58	68
	2.17	.71	69	.64	30	11	39	56	64

<u>CATALYST DESCRIPTIONS</u>	<u>% Ni</u>	<u>% Mo</u>	<u>SUPPORT</u>	<u>S.A.</u>	<u>P.V.</u>	<u>AVG. PORE SIZE</u>
Shell 324	2.7	13.2	Alumina	160	.5	125
Harshaw HT-100	3.8	16.8	1.5% SiO <sub>2</sub> , Alumina	175	.54	123 A
Harshaw HT-500	3.17	15.5	Alumina	210	.49	93 A
Cyanamid HDS9A	3-4	17.5 - 18.5	Alumina	-	-	-
Houdry HR811	3	15	Alumina	7300	-	-

CONVERSION OF SOLVENT REFINED COAL TO DISTILLATE FUELS - G. R. Hass

SUMMARY OF BATCH CATALYST SCREENING TESTS

Table II-I appended to this report, summarizes heteroatom removal data and ASTM distillation data for all batch tests made with PAMCO's Solvent Refined Coal (SRC) Vacuum Flash Feed. All batch tests were made previous to this quarter but complete ASTM Distillation data has not been previously reported. Batch tests were made in a Parr rocking autoclave of 500 ml internal volume. Batch run conditions were as follows:

Vacuum Flash Feed Charged	= 200 gr.
Catalyst Charged	= 25 ml
Initial Hydrogen Pressure	= 2500 psig
Run Temperature	= 420 <sup>o</sup> C + 5 <sup>o</sup> C
Residence Time	= 1 hour at run temp.

From Table II-I it can be seen that Shell 324-E-1/16" catalyst (2.7% Ni, 13.2% Mo on alumina) and MSU STK-14-E-1/16" (17.4% MoO<sub>3</sub>, 2.3% NiO, 1.8% CuO on alumina) gave the best desulfurization in a batch run. It can also be seen that Harshaw Co-Mo-0401-T-1/8" (3% CoO, 9% MoO<sub>3</sub> on alumina) and MSU STK-8-1-E-1/16" (8.6% MoO<sub>3</sub>, 1.4% NiO, 11% WO<sub>3</sub> on alumina) gave the best denitrogenation in a batch run.

SUMMARY OF CONTINUOUS FIXED BED REACTOR TESTS

Table II-II summarizes heteroatom removal data and ASTM Distillation data for continuous fixed bed reactor tests made with PAMCO's SRC Vacuum Flash Feed. Runs numbered C-1 through C-8 were completed previous to this quarter and runs numbered C-9 through C-19 were made during this quarter. These continuous fixed bed reactor runs were made at the following conditions unless specifically noted:

Pressure = 1000 psig  
Temperature = 450°C + 5°C  
H<sub>2</sub> to oil ratio = 10000 SCF/BBL  
Catalyst bed = 60 ml catalyst diluted with 60 ml  
Denstone 1/8" support  
LHSV = 1.0, 1.5, 2.0 hr<sup>-1</sup>

From Table II-II, it can be seen that the best desulfurization, averaged over the three space velocities tested, was obtained with Cyanamid HDS-20AE1/16" (5.0% CoO, 16.2% MoO<sub>3</sub>, on alumina) and Shell 344 E1/16" (2.4% CoO, 9.9% MoO<sub>3</sub> on alumina). The best denitrogenation, over the space velocities tested, was obtained with M.S.U. STK-5-2-2 (.26% NiO, 1.15% CoO, 13.1% MoO<sub>3</sub> on Ketjen -003-E-1/16" support which contains 1.33% SiO<sub>2</sub>).

#### DETAILED RESULTS

Figures 2-1 through 2-3 are plots of percent sulfur in product versus space time (LHSV<sup>-1</sup>). Figure 2-1 is a plot of the best commercial cobalt-molybdate catalysts. Figure 2-2 is a plot of the best commercial nickel-molybdate catalysts. Figure 2-3 is a plot of the best commercial nickel-tungstate catalysts. Figures 2-4 through 2-6 are plots of percent nitrogen in product versus space time (LHSV<sup>-1</sup>). Also plotted on these graphs for comparison purposes, are two catalysts made in our laboratory, namely: MSU-STK-5-2-1 and MSU-STK-5-2-2. Figure 2-4 is a plot of the best commercial cobalt-molybdate catalysts. Figure 2-5 is a plot of the best commercial nickel-molybdate catalysts. Figure 2-6 is a plot of the best nickel-tungstate catalysts. It is very important to note on Figures 2-1 through 2-6 that the data for space time equal to .67 corresponding to a space velocity equal to 1.5 appears to be much better than the other two points on the graphs. This is consistent for almost all the continuous runs and can be attributed to the fact that this is the first steady state sample taken from the reactor while the catalyst is still operating at high initial activity.

Figures 2-7 through 2-11 are plots of ASTM Distillations (D-86) of the three best commercial catalysts and the two best Montana State University catalysts tested to date.

Continuous Run C-14 was made using Harshaw H T-400 E-1/16" catalysts for the purpose of making enough product so it could be used as feedstock for a second pass over the catalyst. C-14 was also run at lower temperature (400°C). Harshaw HT-400-E-1/16" performed well at 450°C, as can be seen in Table II-II, Run C-4. Desulfurization at the lower temperature remained fairly consistent over the twelve hours of the run. Denitrogenation at the lower temperature was good during one initial three hours of the run (C-14-1), but dropped off to zero as the catalyst lined out. Run C-14 thus gave us some good temperature study data which will be useful in the future.

#### CONCLUSIONS FROM THIS QUARTER'S WORK

1. Cyanamid HDS-20A-1/16" Trilobe gave the best average desulfurization over the space velocities tested.
2. MSU-STK-5-2-2-E-1/16" gave the best average denitrogenation over the space velocities tested.
3. The temperature of the runs is extremely critical.

#### FORECAST OF FUTURE WORK

Catalyst testing in the continuous fixed bed reactor will continue. A series of nickel oxide-cobalt oxide-molybdenum trioxide catalysts have been prepared on five different commercial supports. These catalysts will be tested in order to determine any support effects on catalysis. Another series of five cobalt oxide-nickel oxide-tungstate catalysts have been prepared to determine the

effects of cobalt and nickel loadings on catalysis.

Debugging of a gas chromatographic system for use in off-gas analysis from our reactors will continue. If this can be completed, we will be able to complete the mass balance around our continuous reactors.

A linear elution adsorption chromatographic system is being set up to analyze for asphaltene, heavy aromatic, light aromatic, and saturate concentrations in products. When this is in operation, it will be a very useful tool for figuring out what each catalyst is doing to the feedstock.

TABLE II-I

DATA SUMMARY OF BATCH RUNS ON S R C VACUUM FLASH FEED

RUN	CATALYST	% DN*	% DS**	ASTM DISTILLATION			D-86 (VOL %)	YIELD
				IBP-425 <sup>o</sup> F	425-600 <sup>o</sup> F	600-700 <sup>o</sup> F		
26B	Harshaw HT-400-E-1/8"	25.4	34.8	12.0	32.7	23.7	68.4	
27B	Harshaw HT-500-E-1/8"	16.9	33.2	7.8	37.2	19.5	64.5	
28B	Ketjen HC-5-E-1/16"	5.8	27.0	8.0	32.0	21.9	61.9	
29B	Ketjen-330-E-3/32"	12.3	37.3	9.8	35.2	21.2	66.2	
30B	Harshaw Co-Mo-0401-T-1/8"	29.5	36.0	9.5	34.0	24.2	67.9	
31B	MSU-STK-6-E-1/16"	1.5	17.7	10.7	36.1	18.3	65.1	
32B	Shell 324-E-1/16"	27.5	75.3	9.1	39.2	27.4	75.7	
33B	Shell 344-E-1/16"	18.8	52.5	8.5	35.4	32.2	76.1	
34B	MSU-STK-5-E-1/16"	20.2	44.1	8.7	35.8	29.2	73.7	
35B	Harshaw Ni-4401-T-1/8"	20.4	35.7	12.0	28.8	25.0	65.8	
36B	MSU-STK-8-2-E-1/16"	21.5	47.7	8.5	36.8	31.8	77.1	
37B	MSU-STK-10-E-1/16"	12.0	28.3	9.4	32.9	26.8	69.1	
38B	MSU-STK-11-E-1/16"	12.0	38.9	8.3	32.5	28.7	69.5	
39B	Harshaw Ni-4303-1/8"	19.4	42.1	8.8	31.7	25.6	67.05	
40B	MSU-STK-14-E-1/16"	10.2	50.8	8.0	34.0	34.2	76.2	
41B	MSU-STK-9-E-1/16"	14.9	8.7	5.7	34.6	24.3	64.6	
42B	MSU-STK-12-E-1/16"	16.1	46.7	8.7	34.3	24.7	67.7	
43B	MSU-STK-13-E-1/16"	21.7	21.0	8.9	32.8	24.5	66.2	
44B	MSU-STK-8-1-E-1/16"	29.4	15.7	8.1	36.5	25.9	70.5	

\* % DN - Weight Percent Denitrogenation

\*\* % DS - Weight Percent Desulfurization

NOTE: This sample of S R C Vacuum Flash Feed contained .644% Sulfur and 1.30% Nitrogen.

TABLE II-II

## DATA SUMMARY OF CONTINUOUS RUNS ON S R C VACUUM FLASH FEED

RUN	CATALYST	LHSV*	% DN**	% DS***	ASTM DISTILLATION D-66 (VOL %)			
					IBP-425 <sup>0</sup> F	425-600 <sup>0</sup> F	600-700 <sup>0</sup> F	YIELD
C-1	Shell 324-E-1/16"	1.0	38.8	70.5	13.6	41.2	22.1	76.9
		1.5	15.4	61.8	7.3	35.5	31.4	74.2
		2.0	8.5	49.5	7.8	33.8	27.3	68.9
C-3	Harshaw Co-Mo-0401-T-1/8"	1.0	19.2	54.2	8.3	41.5	26.6	76.4
		1.5	12.7	59.9	8.0	36.6	28.2	72.8
		2.0	3.8	38.8	7.9	35.4	31.2	74.5
C-4	Harshaw HT-400-E-1/16"	1.0	33.8	67.1	11.9	39.5	24.2	75.6
		1.5	32.3	53.9	8.5	41.6	28.0	78.1
		2.0	26.2	55.3	8.2	37.6	28.0	73.8
C-5	Harshaw HT-4-0-E-1/8"	1.0	18.8	62.3	8.3	38.4	30.5	77.2
		1.5	20.5	56.4	8.2	36.6	34.3	79.2
		2.0	10.1	34.3	5.6	33.0	29.0	67.6
C-6	Harshaw HT-500-E-1/8"	1.0	18.5	53.1	7.9	38.8	31.4	78.1
		1.5	20.0	58.8	7.9	37.5	26.3	71.7
		2.0	13.1	53.6	5.7	36.0	33.3	75.0
C-7	Shell 344-E-1/16"	1.0	31.1	73.9	12.4	38.9	23.3	74.6
		1.5	37.0	61.2	13.5	39.0	24.2	76.7
		2.0	17.7	65.2	8.0	38.3	28.9	75.2
C-8	Harshaw Ni-4401-T-1/8"	1.0	15.2	66.3	7.7	35.6	30.01	73.4
		1.5	17.8	38.2	8.1	34.6	31.5	74.2
		2.0	13.5	41.5	1.3	38.2	32.7	72.2
C-9	Cyanamid HDS-20A	1.0	18.4	62.5	10.2	47.0	26.7	83.9
		1.5	16.7	70.8	9.7	44.6	27.1	81.4
		2.0	11.0	69.4	9.0	44.5	23.8	77.3
C-10	Cyanamid HDS-9A	1.0	21.1	61.1	7.7	49.3	24.6	81.6
		1.5	26.3	62.5	12.5	51.0	17.7	81.2
		2.0	21.1	61.1	8.2	47.3	25.0	80.5

continued....

TABLE II-II - Continued

## DATA SUMMARY OF CONTINUOUS RUNS ON S R C VACUUM FLASH FEED

RUN	CATALYST	LHSV*	% DN**	% DS***	ASTM DISTILLATION D-66 (VOL %)			YIELD	
					IBP-425°F	425-600°F	600-700°F		
C-11	Harshaw Co-Mo-0603-T-1/8"	1.0	13.2	54.2	13.0	43.0	21.7	77.7	
		1.5	15.4	45.8	12.4	46.7	21.8	80.9	
		2.0	4.4	54.9	13.0	57.6	20.9	75.5	
C-12	Harshaw Ni-4301-T-1/8"	1.0	9.6	61.1	12.0	39.6	23.0	74.6	
		1.5	10.5	51.4	10.4	41.3	28.4	80.1	
		2.0	2.2	48.6	9.9	40.5	22.1	72.5	
C-13	Harshaw Ni-4303-T-1/8"	1.0	11.4	53.5	10.2	45.9	25.7	81.8	
		1.5	15.8	55.6	10.2	44.7	24.9	79.8	
		2.0	11.4	48.6	8.8	43.7	24.1	76.6	
C-14	Harshaw HT-400-E-1/16"								
		C-14-1	2.0	20.2	54.9	4.7	42.7	22.3	69.7
		C-14-2	2.0	1.8	43.1	6.3	38.2	27.2	71.7
		C-14-3	2.0	-0-	47.2	9.5	38.5	24.8	72.8
		C-14-4	2.0	-0-	41.7	6.5	38.5	23.4	68.4
C-15	Harshaw HT-100-E-1/12"	1.0	17.1	56.2	10.1	45.3	23.3	78.7	
		1.5	21.5	51.4	10.2	45.7	22.7	78.6	
		2.0	6.0	54.2	7.5	43.2	21.3	72.0	
C-16	Harshaw Ni-3250-T-1/8"	1.0	18.0	21.5	7.5	43.5	22.5	73.5	
		1.5	14.5	16.0	5.1	47.5	21.2	73.8	
		2.0	4.8	18.1	2.7	42.6	23.3	68.6	
C-17	Harshaw Ni-1601-T-1/8"	1.0	30.3	20.1	5.6	44.0	25.3	74.9	
		1.5	32.9	27.8	6.1	50.7	23.9	80.7	
		2.0	24.1	4.2	7.1	40.5	28.5	76.1	
C-18	MSU-STK-5-2-1-E-1/16"	1.0	32.9	47.9	11.8	42.8	22.6	77.2	
		1.5	39.5	47.9	11.9	43.6	21.2	76.7	
		2.0	29.8	39.6	10.0	40.7	24.9	75.6	

continued...

TABLE II-II - Continued

DATA SUMMARY OF CONTINUOUS RUNS ON S R C VACUUM FLASH FEED

RUN	CATALYST	LHSV*	% DN**	% DS***	ASTM DISTILLATION D-66 (VOL %)			YIELD
					IBP-425 <sup>0</sup> F	425-600 <sup>0</sup> F	600-700 <sup>0</sup> F	
C-19	MSU-STK-5-2-2-E-1/16"	1.0	46.5	56.2	15.4	45.3	25.7	86.4
		1.5	54.8	65.3	14.8	45.6	27.2	88.6
		2.0	35.1	53.5	8.1	44.5	26.3	78.9

- \* LHSV - Liquid Hourly Space Velocity
- \*\* % DN - Weight Percent Denitrogenation
- \*\*\* % DS - Weight Percent Desulfurization

NOTES:   Runs C-1 through C-8   Feedstock contained:   .644% S and 1.30% N  
           Runs C-9 through C-19   Feedstock contained:   .72% S and 1.14% N

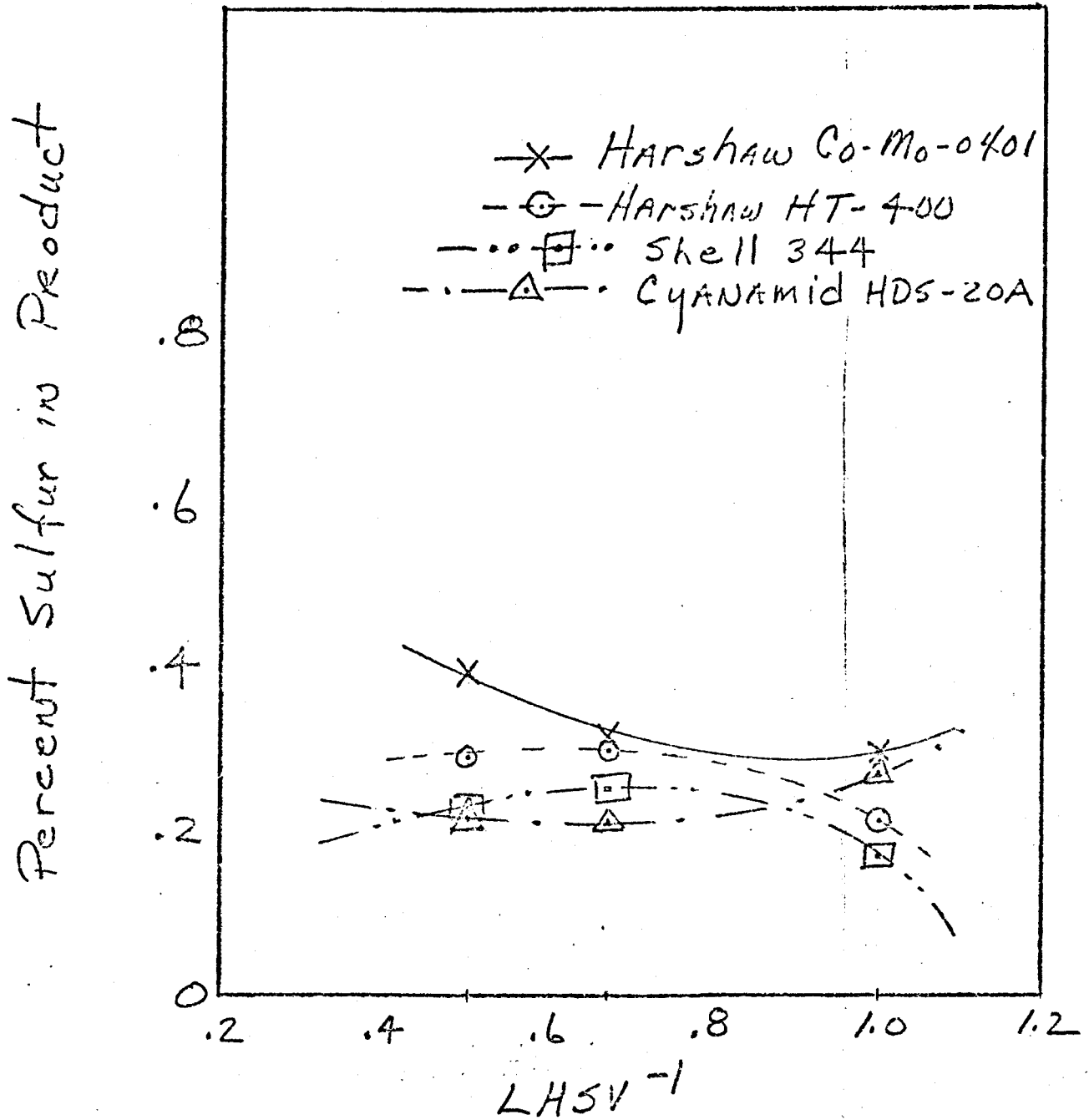


Fig. 2-1 Commercial Co-Mo Catalysts

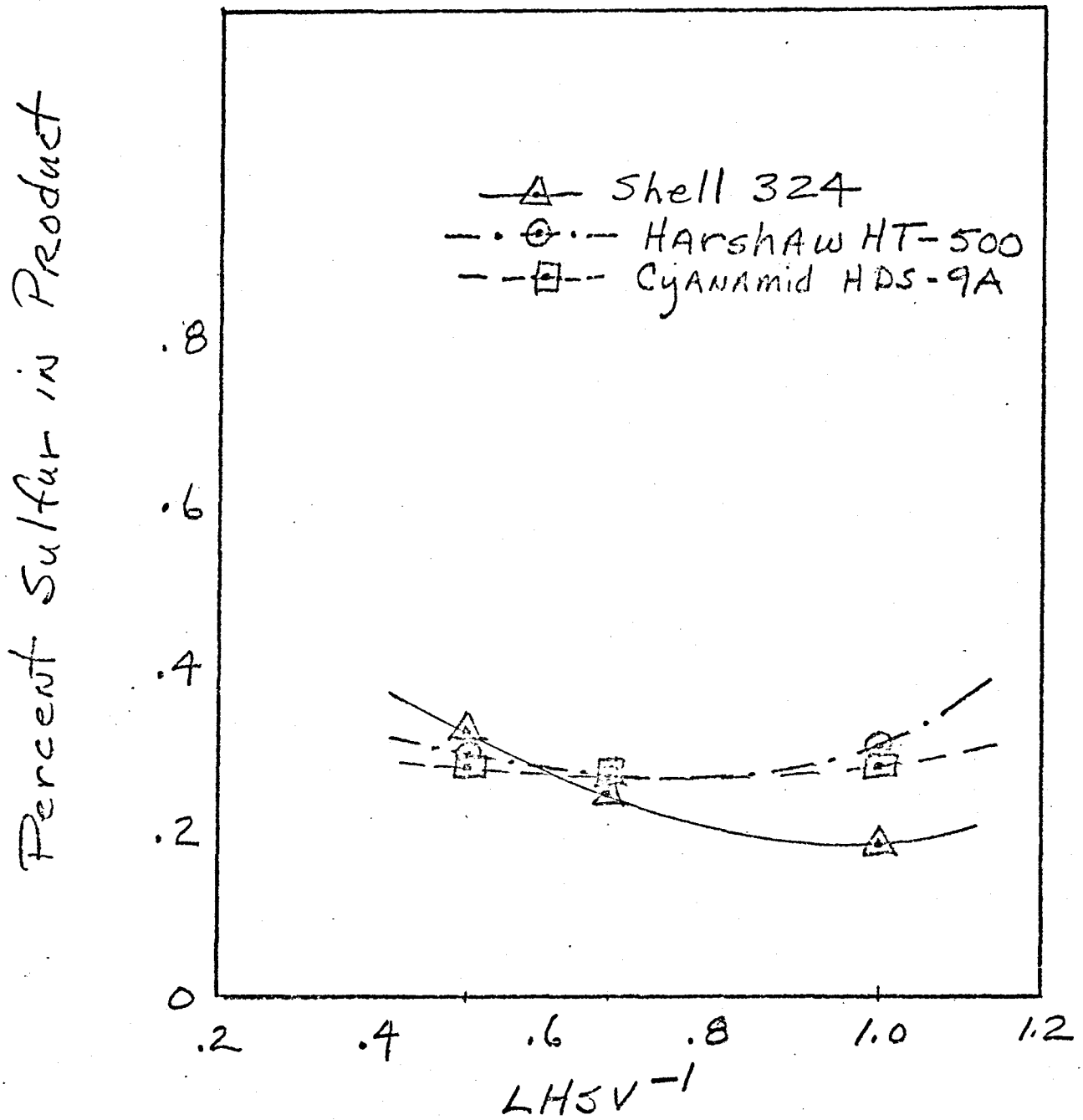


Fig. 2-2 Commercial Ni-Mo Catalysts

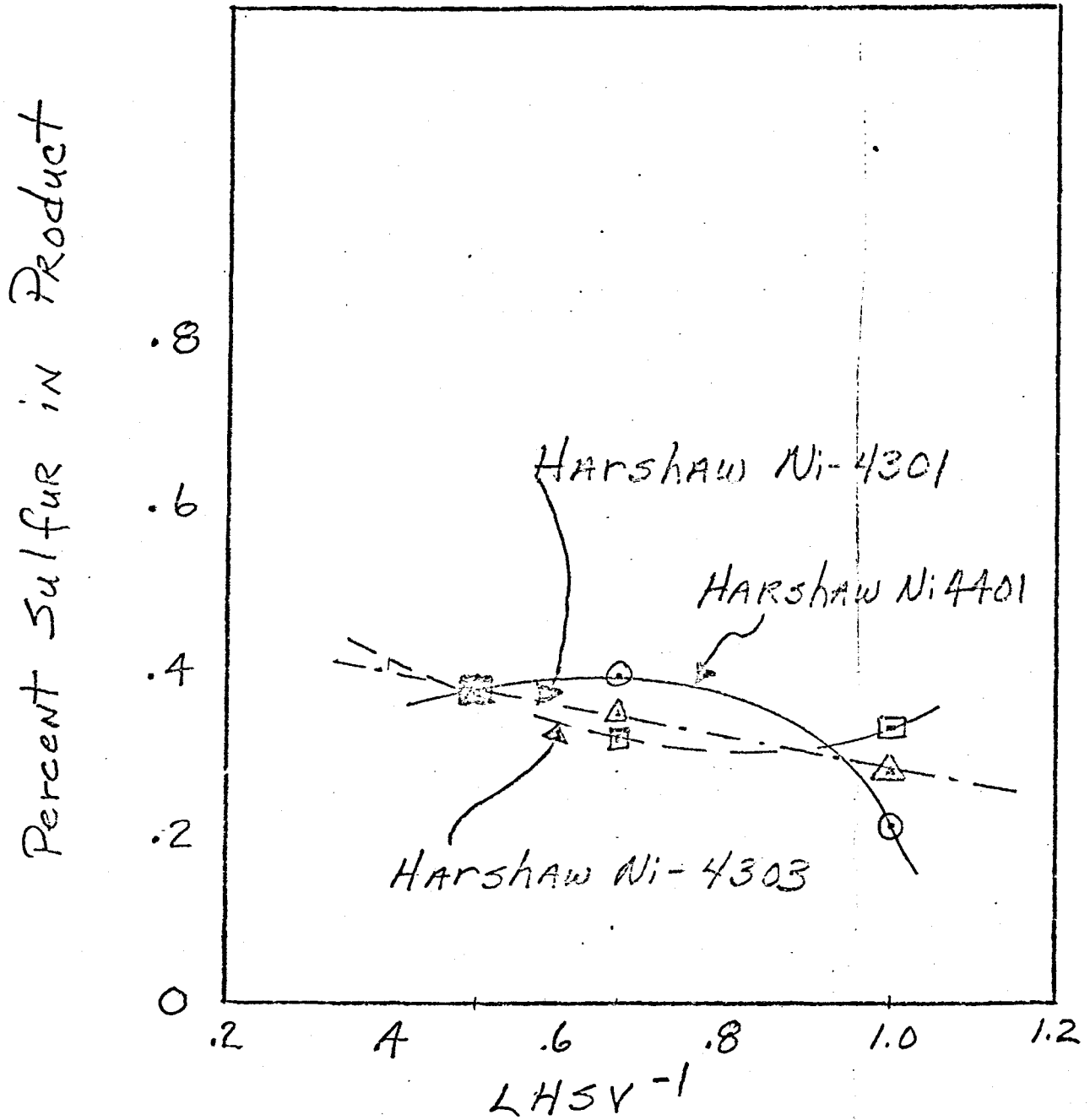


Fig 2-3 Commercial Ni-W Catalysts

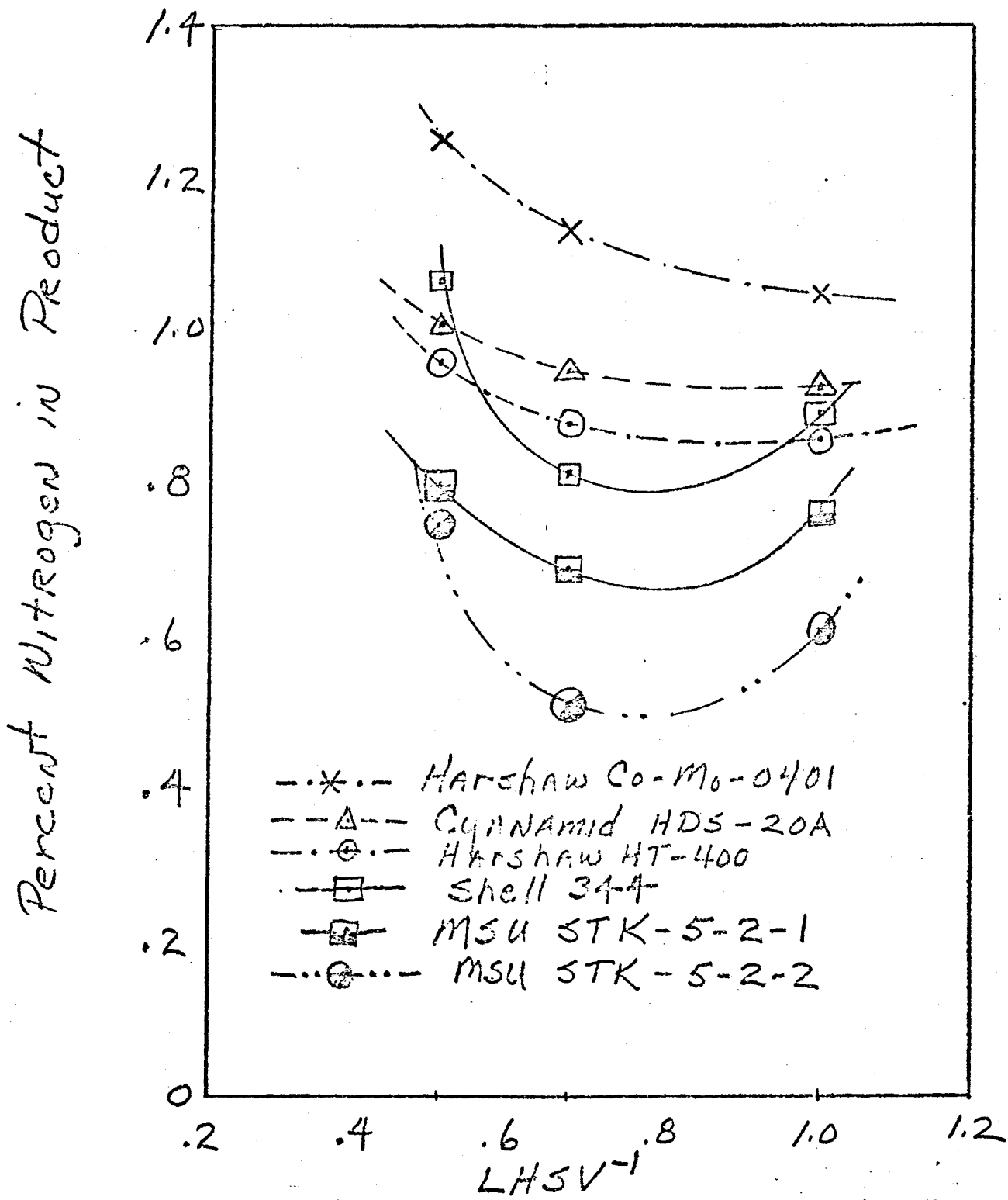
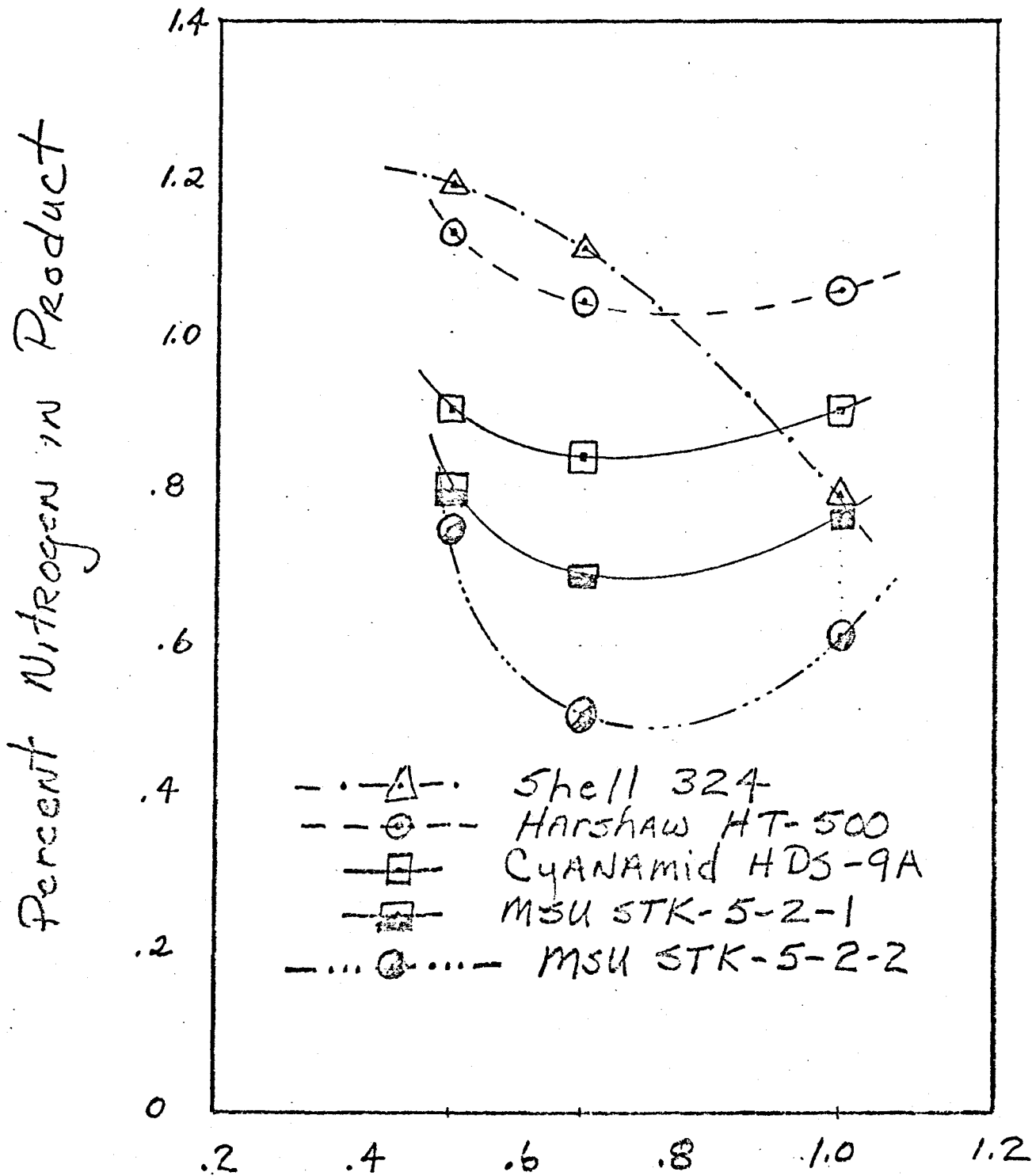


Fig. 2-4 Commercial Co-Mo Catalysts



LHSV-1  
Fig. 2-5 Commercial Ni-Mo Catalysts

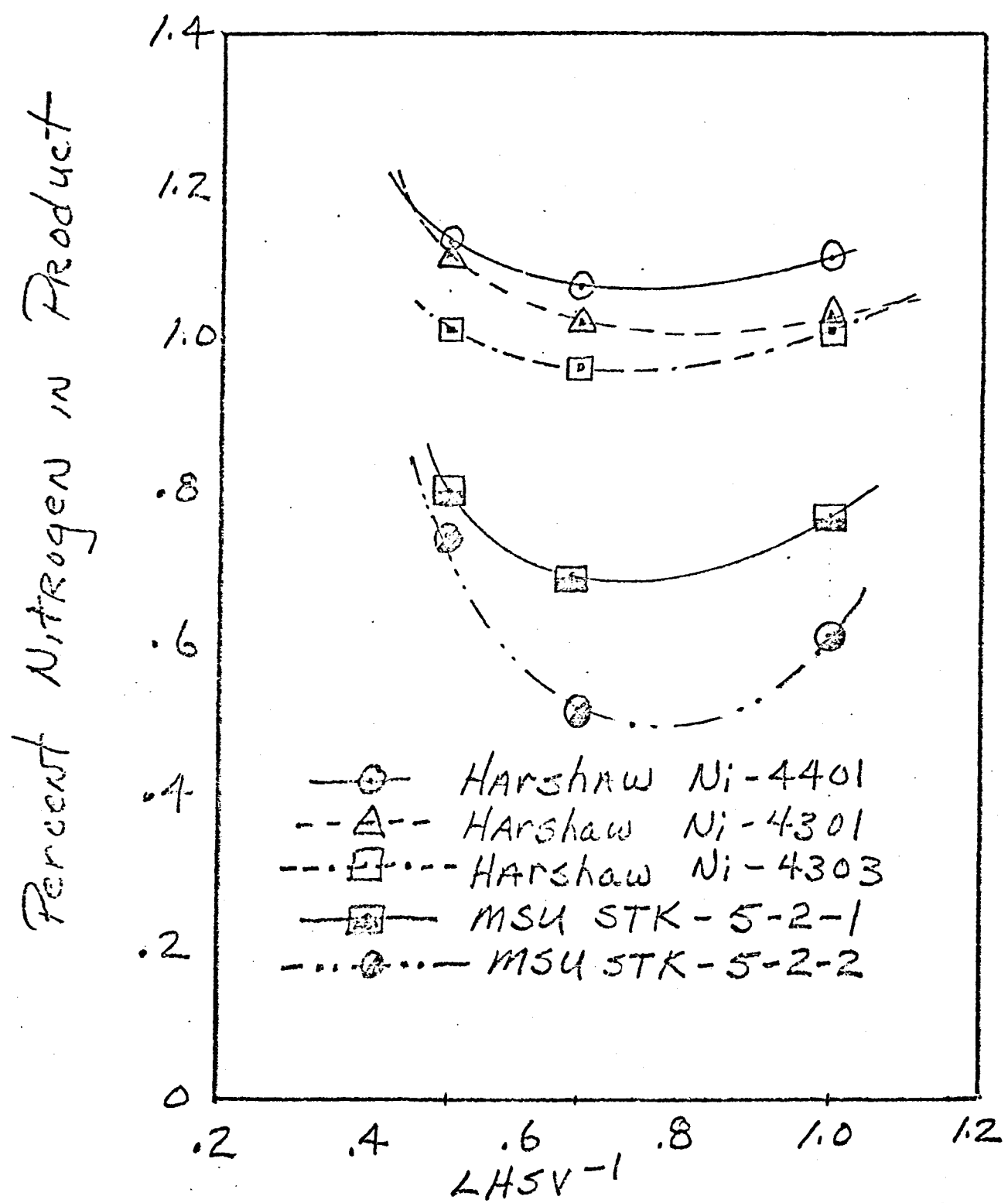


Fig. 2-6 Commercial Ni-W Catalysts

SRC - G. HASS

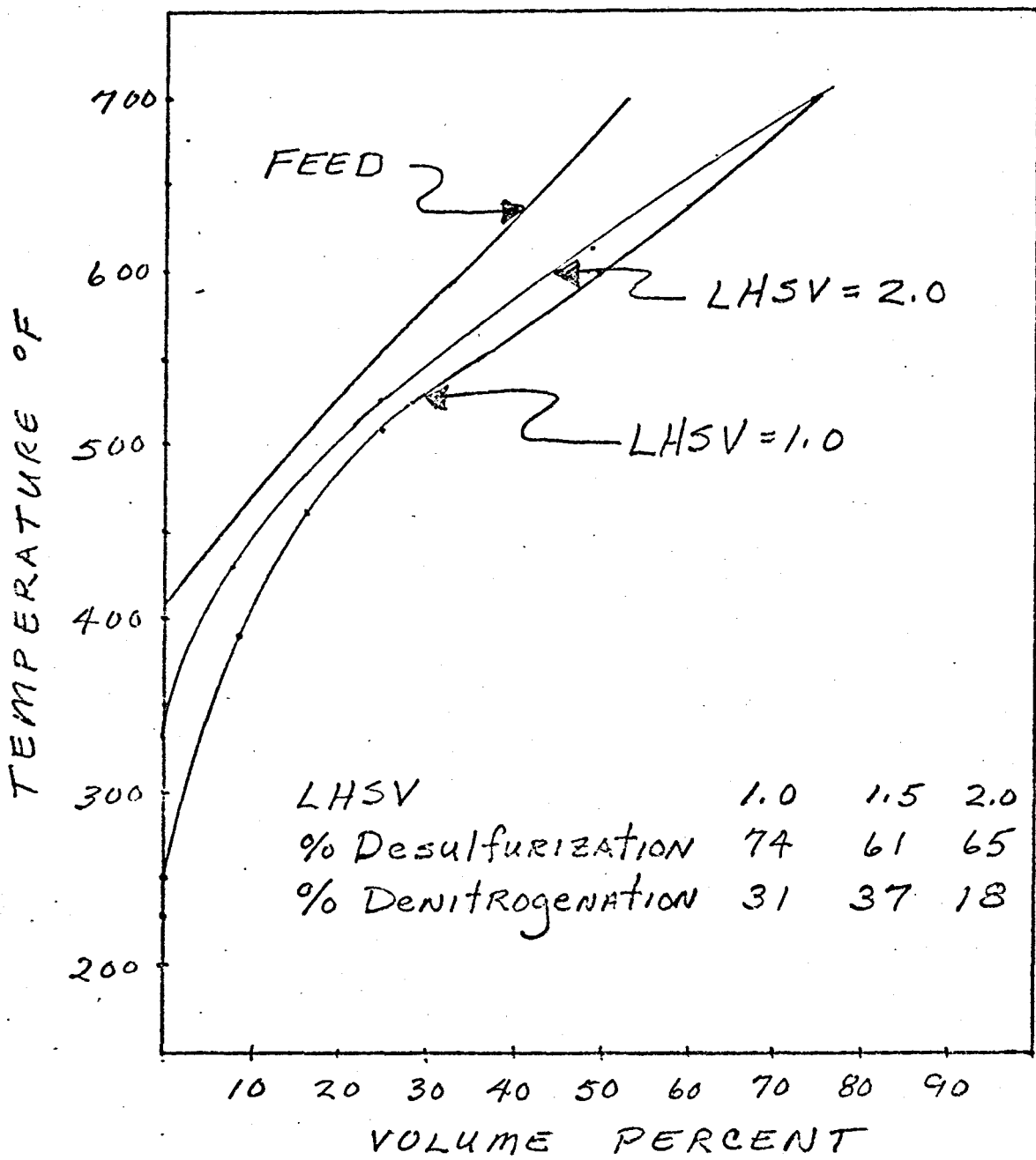


Fig. 2-7 Shell 344 E 1/16"  
2.4% CoO, 9.9% MoO<sub>3</sub> on Al<sub>2</sub>O<sub>3</sub>

SRC - G. HASS

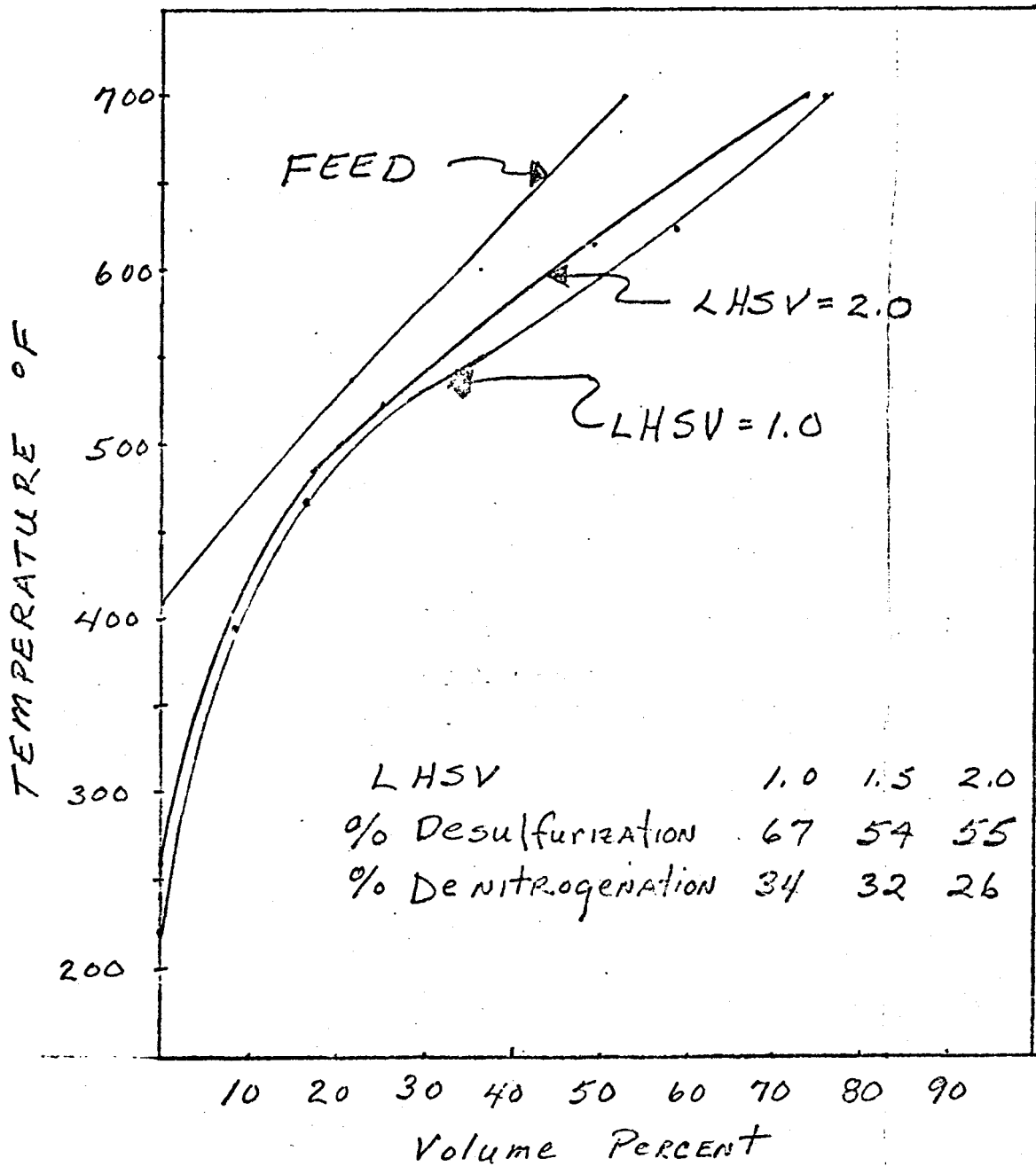


Fig. 2-8 HARSHAW HT-400 E 1/16"  
3% CoO, 15.0% MoO<sub>3</sub> on Al<sub>2</sub>O<sub>3</sub>

SRC - G. HASS

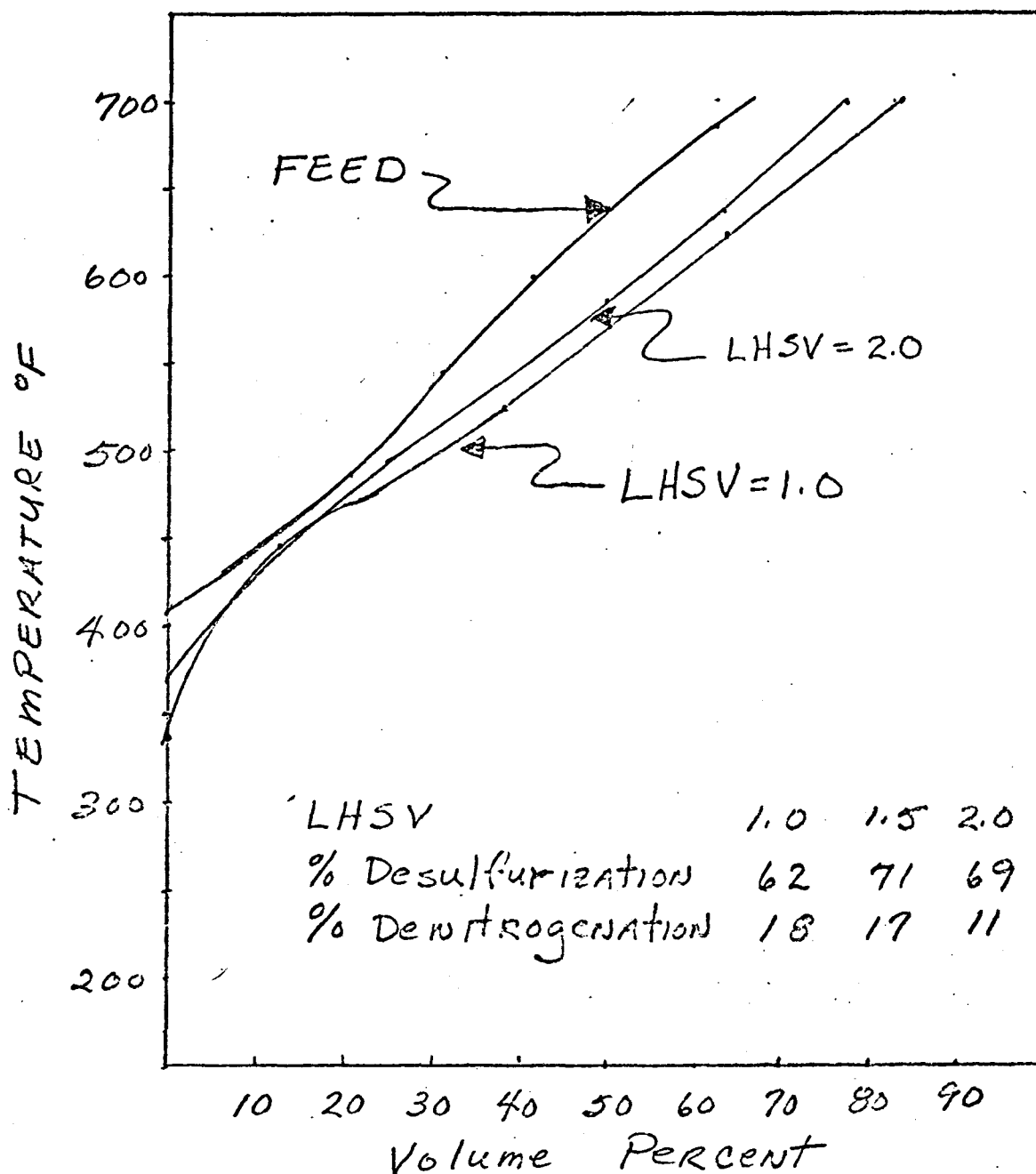


Fig. 2-9 Cyanamid HDS-20A E 1/8"  
5.0% CoO, 16.0% MoO<sub>3</sub> on Al<sub>2</sub>O<sub>3</sub>

SRC - G. HASS

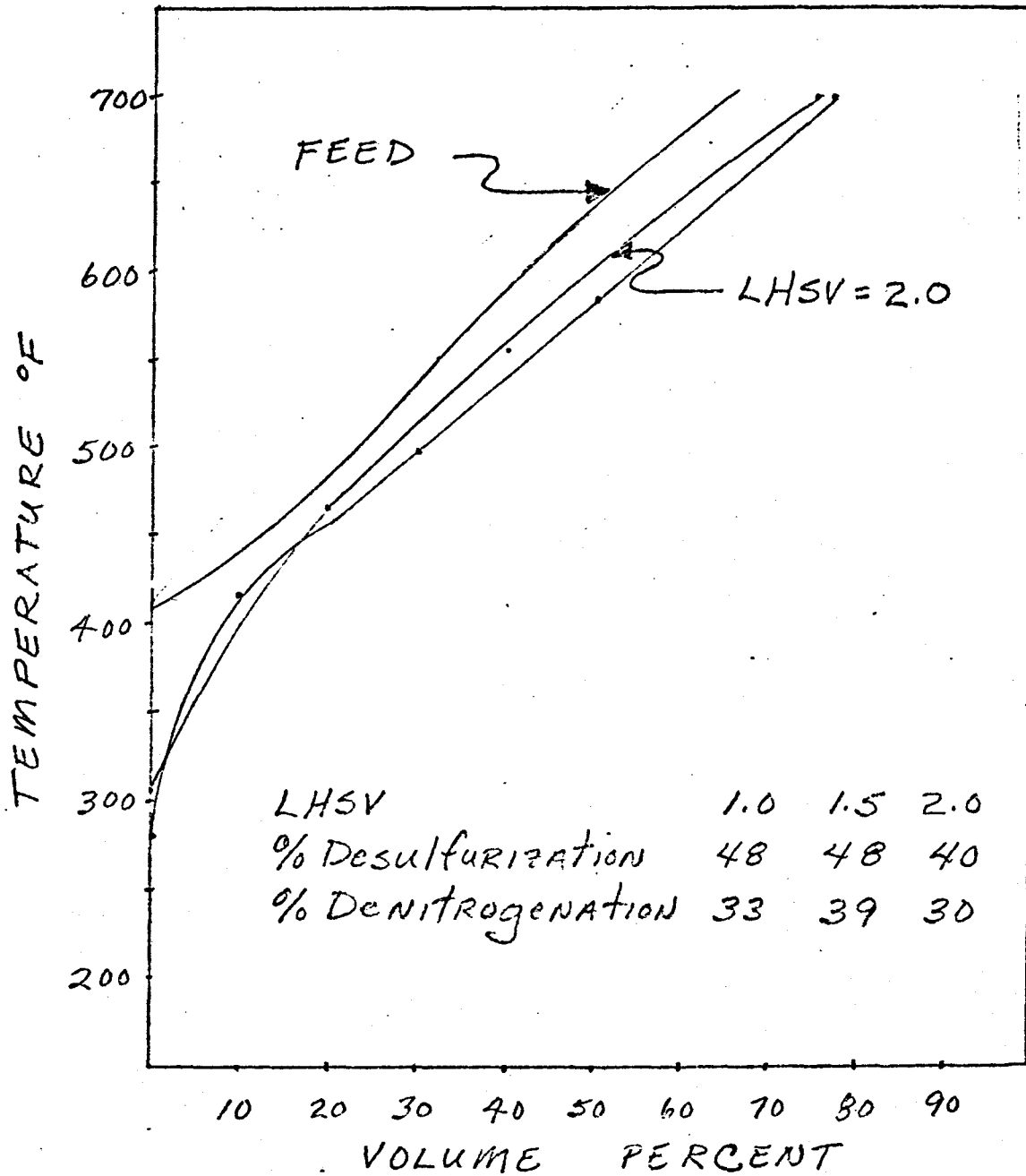


Fig. 2-10 MSU STK-5-2-1 E 1/16"  
.48% NiO, .75% CoO, 23.9% MoO<sub>3</sub>  
ON NORTON 6176 E 1/16 Al<sub>2</sub>O<sub>3</sub>

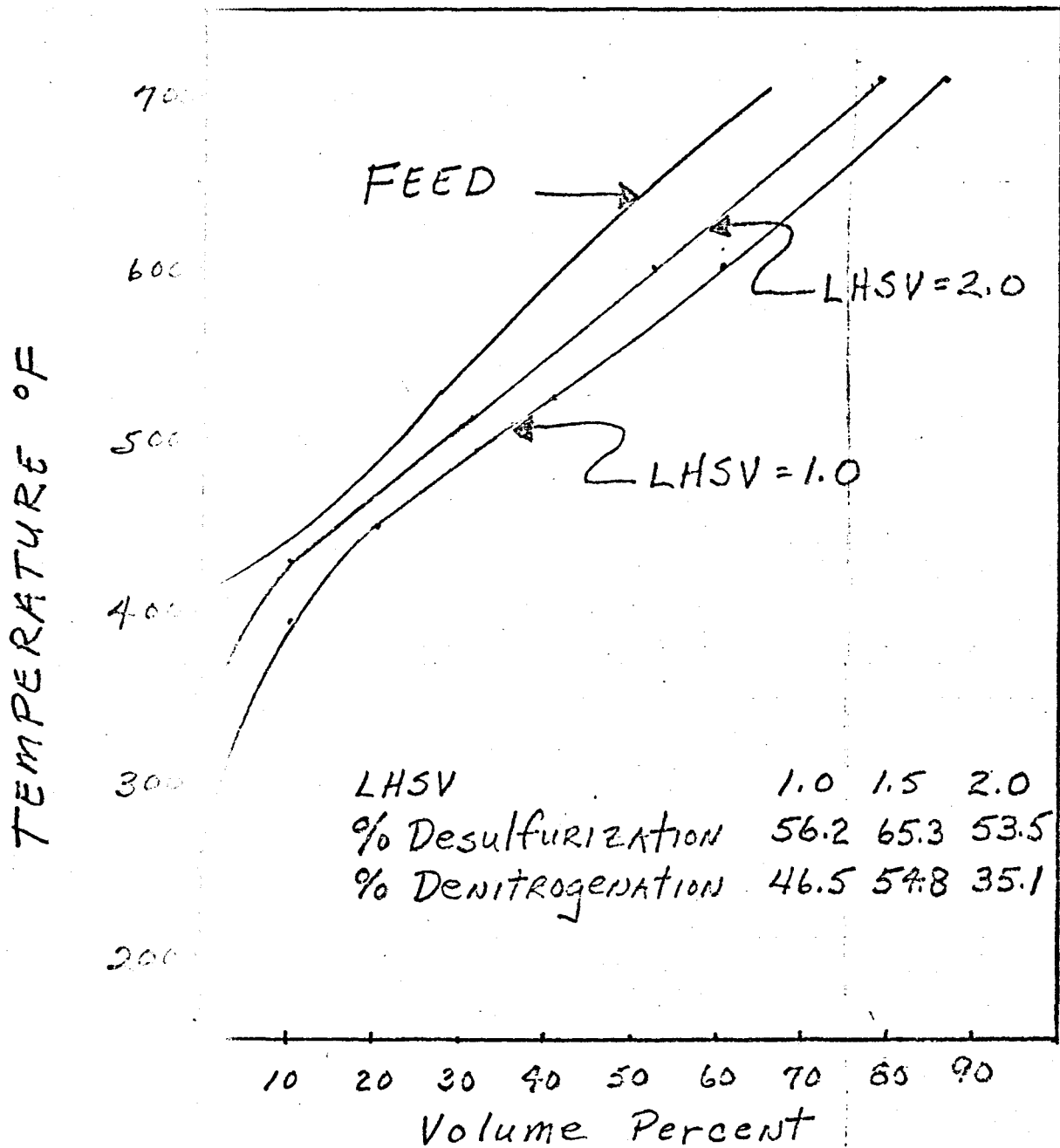


Fig. 1 MSU STK-5-2-2 E 1/16"  
.26% NiO, 1.15% CoO, 13.1% MoO<sub>3</sub>  
ON Ketjen-003 · 1.33% SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>

TABLE II-III  
CATALYST DESCRIPTION

Harshaw CoMo 0401 T 1/8	3% CoO, 9% MoO <sub>3</sub> on silica alumina
Harshaw HT 100	3.8% NiO, 16.8% MoO <sub>3</sub> on silica alumina
Harshaw HT 500	3.2% NiO, 16% MoO <sub>3</sub> on alumina
Harshaw Ni 4401	6% NiO, 19% WO <sub>3</sub> ; 50% SiO <sub>2</sub> , 25% Al <sub>2</sub> O <sub>3</sub>
Harshaw Ni 4301	6% NiO, 19% WO <sub>3</sub> on silica alumina
Harshaw Ni 4303	6% NiO, 19% WO <sub>3</sub> on alumina
Harshaw Ni 1601	3% NiO, 3% CoO, 3% Fe <sub>2</sub> O <sub>3</sub> on alumina
Harshaw Ni 3250	50% Nickel
Harshaw Mo-1201	10% MoO <sub>3</sub> on alumina
Harshaw W 0801	10% WO <sub>3</sub> on alumina
Harshaw Ni 1600	3% CoO, 3% NiO, 3% Fe <sub>2</sub> O <sub>3</sub> on silica alumina
Shell 324	3% NiO, 13% MoO <sub>3</sub>
Shell 344	2.4% CoO, 9.9% MoO <sub>3</sub>
Ketjen HC-5	6.5% NiO, 21% WO <sub>3</sub> on alumina
Ketjen Ketjenfine 330-3E	6.6% NiO, 19.8% WO <sub>3</sub> , 1.2% SiO <sub>2</sub> on alumina
MSU-STK-5	1.2% CoO, 0.4% NiO, 18.2% MoO <sub>3</sub> on alumina
MSU STK-6	1.5% CoO, 1.2% NiO, 18% MoO <sub>3</sub> on alumina
CCI-C20-6	4% CoO, 15% WO <sub>3</sub> on alumina
Harshaw HT 400 E1/16"	3% CoO, 15% MoO <sub>3</sub> , on alumina S.A. = 220 M <sup>2</sup> /g, P.V. = .55cc/g
Houdry, HR 801	3% CoO, 15% MoO <sub>3</sub> , on alumina S.A. = 300 M <sup>2</sup> /g, 1/16" extrudates
Norton 6176 Alumina Supports	Alumina Supports. Al <sub>2</sub> O <sub>3</sub> (>99.85%) Na <sub>2</sub> O (<0.014%) SiO <sub>2</sub> (<0.12%) Fe <sub>2</sub> O <sub>3</sub> (<0.065%) S.A. = 250+20 M <sup>2</sup> /g, P.V. = 0.8-101cc/gr before impregnation.

Continued...

TABLE II-III -continued

CATALYST DESCRIPTION

Cyanamid HDS-20A	5% CoO, 16.2% MoO <sub>3</sub> on alumina, 1/16" Tilobe S.A. = 230 m <sup>2</sup> /g P.V. = .52 cc/g
Cyanamid HDS-9A	3.5% NiO, 18.0% MoO <sub>3</sub> on alumina, S.A. = 300 m <sup>2</sup> /g, 1/8" extrudates
Houdry HR-811	3.0% NiO, 15.0% MoO <sub>3</sub> on alumina, S.A. = 300 m <sup>2</sup> /g, 1/8" extrudates
Harshaw Co-Mo-0603	3% CoO, 12.0% MoO <sub>3</sub> on alumina
M.S.U.-STK-5-2-1	.48% NiO, 23.9% MoO <sub>3</sub> , .75% CoO on Norton 6176 support.
M.S.U.-STKk-5-2-2	.26% NiO, 1.15% CoO, 13.1% MoO <sub>3</sub> , on Ketjen- 003-1.5E support which contains 1.33% SiO <sub>2</sub> .01% NA <sub>2</sub> O, Balance Al <sub>2</sub> O <sub>3</sub> S.A. = 240 m <sup>2</sup> /g P.V. = .7 cc/g

CATALYTIC HYDROGENATION OF SYNTHOIL - M. D. Anderson

SUMMARY OF PREVIOUS WORK

To this date, a total of 35 bomb runs have been made on Synthoil using 26 commercially prepared catalysts and three M.S.U. catalysts. Synthoil has been tested with ten commercial catalysts in the continuous reactor. In the continuous reactor, Harshaw Catalyst HT-500 gave the best sulfur removal, and Shell 324 Catalyst gave the best hydrocracking and nitrogen removal. The best M.S.U. catalyst tested in the bomb is STK-10. The properties of these catalysts are given in Table 3-I.

SUMMARY OF PROGRESS DURING THIS REPORTING PERIOD

During this quarter, two bomb runs and two continuous runs were made on Synthoil. Bomb runs were made using two MSU catalysts, MSU-STK-12 and MSU-STK-14. Two commercial catalysts, Shell 324 and Shell 344, were tested in the continuous reactor. All of the catalysts tested were sulfided.

DISCUSSION OF BOMB RUNS

Two MSU catalysts were tested in a 500 ml. Parr rocking autoclave under the conditions given below:

Synthoil charged	= 150 ml.
Catalysts charged	= 25 ml.
Initial Hydrogen pressure	= 2000 psig
Run Temperature	= $450^{\circ}\text{C} \pm 5^{\circ}\text{C}$
Run Time	= 1 hour

The two catalyst tested were MSU-STK-14 and MSU-STK-12 which are described in Table 3-I. The compositions of the two catalysts are approximately the same with the exception that MSU-STK-14 contains 1.8% CuO while MSU-STK-12 contains 1.7% ZnO.

Table III-II gives the results of the ASTM distillations from runs B33, B34 and B35 along with the distillation results of untreated Synthoil. From this Table, it can be seen that STK-14 cracked the Synthoil only slightly better than MSU-STK-12. The highest degree of hydrocracking achieved in the bomb to date was given by MSU-STK-10 catalyst in Run B33.

Table III-III presents the bomb run heteroatom removal data for two of the three MSU catalysts tested. The product from run B34 was inadvertently rendered useless for heteroatom analysis.

### DISCUSSION OF CONTINUOUS RUNS

Two commercial catalysts were tested in the continuous fixed bed reactor at the following conditions:

Pressure	=	800 psig
Temperature	=	450°C + 5°C
H <sub>2</sub> :Synthoil ratio	=	10,000 SCf/bbl
Catalyst bed	=	70 ml catalyst diluted with 70 ml. Denstone 1/8" inert support material
LHSV	=	1.23, 2.0

The first run (CR1) was made using Shell 344 Catalyst (2.4% Co, 9.9% Mo on alumina) at a liquid hourly space velocity (LHSV) of 2.0. Run CR2 was made using Shell 324 catalyst (1.7% Ni, 13.2% Mo on alumina) at space velocities of 1.23 and 2.0. The best results were obtained at the lowest space velocity.

Table III-IV presents the ASTM distillation results for the products of the two continuous runs. Figure 3-1 is a plot of the ASTM distillation data for Run CR2 which shows that a higher degree of hydrocracking was achieved at the lower space velocity. Products CR2-2 and CR2-3 were made at approximately the same conditions but different amounts were distilled, 20 ml and 50 ml, to determine if varying the amount of the sample distilled would affect the results. Figure 3-1 indicates that the two distillations give essentially the same results.

Table III-V presents the continuous run heteroatom removal data. The highest denitrogenation to date (43%) was achieved by Shell 324 catalyst at a space velocity of 1.23. The sulfur removal for this run ( 36%) was relatively low, however, and does not seem to correlate with the desulfurization that was achieved in the bomb for this catalyst (55%).

#### CONCLUSIONS FROM THIS QUARTERS WORK

1. The best MSU catalyst tested to date is MSU-STK-10 which gave the best hydrocracking of all the catalysts that were tested in the bomb.
2. The best hydrocracking and nitrogen removal in the continuous reactor were given by Shell 324 catalyst.

#### FORECAST OF FUTURE WORK

Batch testing of catalysts will continue to screen catalyst activity.

Runs will be made to try to determine the effects of catalyst pore volume, surface area, and mean pore diameter on catalyst activity.

TABLE III-I  
CATALYST PROPERTIES

RUN. NO.	CATALYST
9-1 & B25	Harshaw HT-500 (1/8" extrusions) 3% NiO, 15.5% MoO <sub>3</sub> on alumina, Surface area - 210 m <sup>2</sup> /g Pore volume - .44 cc/g
B33	MSU-STK-10 (1/16" extrusions), 12.0% MoO <sub>3</sub> , 1.7% CoO, 2.6% NiO, .9% Fe <sub>2</sub> O <sub>3</sub> on alumina.
B34	MSU-STK-14 (1/16" extrusions) 17.4% MoO <sub>3</sub> , 2.3% NiO, 1.8% CuO on alumina
B35	MSU-STK-12 (1/16" extrusions) 18% MoO <sub>3</sub> , 2% NiO, 1.7% ZnO on alumina
CR1	Shell 344 (1/16" extrusions) 2.4% Co, 9.9% Mo on alumina, Surface area = 195 m <sup>2</sup> /g, Pore volume = .6 cc/g
CR2	Shell 324 (1/16" extrusions) 1.7% Ni, 13.2% Mo on alumina, Surface area - 160 m <sup>2</sup> /g, Pore volume - .5 cc/g

NOTE: All MSU catalysts have been prepared on Norton 6176 alumina support (99.85% Al<sub>2</sub>O<sub>3</sub>, .014% Na<sub>2</sub>O, .12% SiO<sub>2</sub>, .065% Fe<sub>2</sub>O<sub>3</sub>, Pore volume - .95 cc/g, surface area - 250 ± 20 m<sup>2</sup>/g).

TABLE III-II  
BOMB RUN DISTILLATE YIELDS

RUN NO.	CATALYST	% NAPHTHA IBP-425 <sup>o</sup> F	% FUEL OIL 425-600 <sup>o</sup> F	% GAS OIL 600-700 <sup>o</sup> F	% RESIDUE 700 <sup>o</sup> F	TOTAL VOL %
-	Synthoil	4	20	20	56	44
B33	MSU-STK-10	11	25	31	33	67 <sup>(a)</sup>
B34	MSU-STK-14	11	23	28	38	62
B35	MSU-STK-12	9	24	27	40	60

(a) This is the highest degree of hydrocracking achieved to date.

TABLE III-III  
BOMB RUN HETEROATOM REMOVAL (a)

RUN NO.	CATALYST	% deS (b)	% de N (c)
B33	MSU-STK-10	36	23
B34	MSU-STK-14	(d)	(d)
B35	MSU-STK-12	17	22

- (a) Shell 324 has provided the best desulfurization (55%), and Harshaw HT-500 has provided the best denitrogenation (33%) to date.
- (b) Weight percent desulfurization based on Synthoil containing .44 weight percent sulfur.
- (c) Weight percent denitrogenation based on Synthoil containing 1.06 weight percent nitrogen.
- (d) Product rendered useless for heteroatom analysis.

TABLE III-IV  
CONTINUOUS RUN DISTILLATE YIELDS

RUN NO.	CATALYST	% NAPHTHA IBP-425 <sup>o</sup> F	% FUEL OIL 425-600 <sup>o</sup> F	% GAS OIL 600-700 <sup>o</sup> F	% RESIDUE 700 <sup>o</sup> F	TOTAL VOL %
-	Synthoil	4	20	20	56	44
CR1-2	Shell 344	15	29	26	30	70
CR2-1	Shell 324	12	36	29	23	77 <sup>(a)</sup>
CR2-2	Shell 324	7	32	26	35	65
CR2-3	Shell 324	11	29	22	38	62

(a) This is the highest degree of hydrocracking achieved to date.

TABLE III-V  
CONTINUOUS RUN HETEROATOM REMOVAL (a)

RUN NO.	LHSV	H <sub>2</sub> :OIL RATIO, SCF/BBL	% de S (b)	% de N (c)
CR1-2	2.1	10,000	24	25
CR2-1	1.23	10,000	36	43
CR2-2	2.0	10,000	32	35
CR2-3	2.0	10,000	30	35

- (a) Harshaw HT-500 has provided the best desulfurization (70%), and Shell 324 has provided the best denitrogenation (43%) to date.
- (b) Weight percent desulfurization based on Synthoil containing .44 weight percent sulfur.
- (c) Weight percent denitrogenation based on Synthoil containing 1.06 weight percent nitrogen.

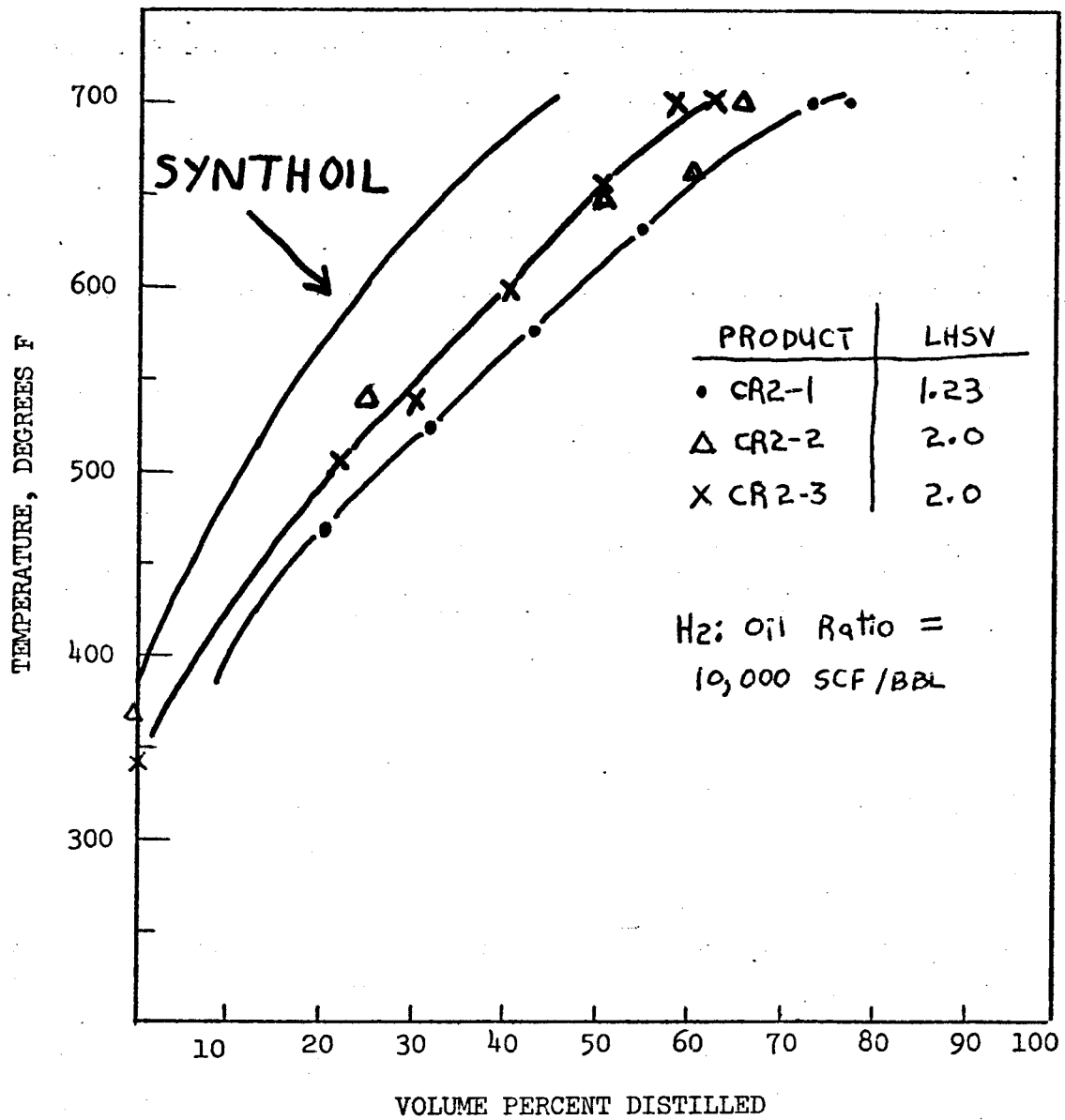


FIGURE 3-1 - ASTM DISTILLATION CURVES FOR  
RUN CR2 USING SHELL CATALYST  
324

PICTORIAL PROGRESS REPORT

Build  
Equipment

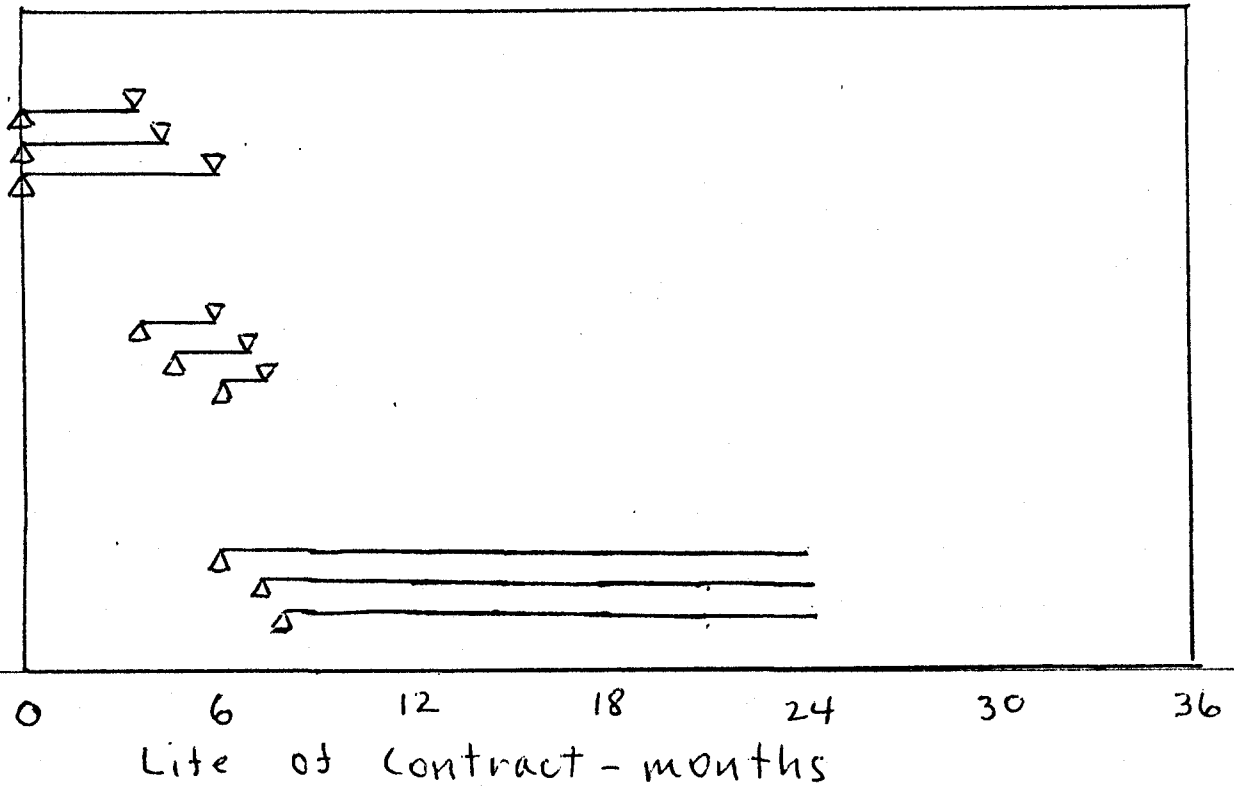
COED  
SYNTHOIL  
S R C

Test Continuous  
Equipment

COED  
SYNTHOIL  
S R C

Catalyst  
Development

COED  
SYNTHOIL  
S R C



G &amp; C

## PROJECT FINANCIAL REPORT

NO. 789

6/30/77

DEPT 6131 BERG

DISTILLATE FUELS

BEGIN DATE 07-01-75

END DATE 6-19-78

	CURRENT MO.	TOTAL TO DATE	AMT. AUTH.
RECEIPTS	1255.03	75501.72	155932.00

EXPENDITURES			ENCUMBRANCES	TOTAL COMMITTED	BUDGET
SALARY AND WAGES	1536.63	37647.16		37647.16	68657.
BENEFITS	9.67	1549.78		1549.78	4255.
REP & MAINT	423.42	431.42		431.42	
TRAVEL		1349.29		1349.29	3000.
COMMUNICATIONS		278.32		278.32	
COMPUTER					3000.
SUPPLIES	1098.43	12751.24		12751.24	12750.
SUBCONTRACT		150.97		150.97	
EQUIPMENT	3053.95	8473.11		8473.11	12700.
AWARDS		1450.15		1450.15	6000.
INDIRECT CHARGES	1057.82	26814.62	18755.38	45570.00	45570.
TOTAL	7179.92	90996.06	18755.38	109651.44	155932.
BALANCE	CASH -15394.34		FREE BALANCE	46280.56	

DETAIL TRANSACTION	DATE	NUMBER	DESCRIPTION	AMOUNT	TOTAL
	06-01-77	E006916	TREAS OF U S	1255.03	1255.03
	- -7		HAUGEN MARILYN M	9.00	
	- -7		HASS GARY R	100.00	
	- -7		HENTON LEE M	400.00	
	- -7		KUJAWA STEPHAN T	400.00	
	- -7		ANDERSON MARK D	39.00	
	- -7		ANDERSON MARK D	60.03	
	- -7		CONRAD ELIZABETH A	301.50	
	- -7		HASS DANAE A	213.00	
	06-07-77	J701338	FIN AID WRK STUDY	5.40	
	06-21-77	J701435	APR CWS	8.70	1536.63
	06-07-77	J701338	FIN AID WRK STUDY	.36	
	06-08-77	J701351	MAY MT IA	4.16	
	06-21-77	J701435	APR CWS	.58	
	06-30-77	J701505	JUN MT I A	1.83	

NO. 789

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DETAIL TRANSACTION	DATE	NUMBER	DESCRIPTION	AMOUNT	TOTAL
	06-30-77	J701551	JULY 15 NIA	2.74	9.67
	06-02-77	7409453	SARGENT-WELCH SCI	13.65	
	06-02-77	7409480	VWR SCIENTIFIC	133.00	
	06-01-77	J701308	XEROX REVOL CHEM	287.62	
	06-06-77	7409521	VWR SCIENTIFIC	46.20	
	06-03-77	J701314	CHEM STORES	135.19	
	06-10-77	7409733	VWR SCIENTIFIC INC	83.36	
	06-14-77	J701394	TECHNICAL SERVICE	18.00	
	06-15-77	7409984	AMER CHEMICAL SOC	16.50	
	06-15-77	7409985	SARGENT-WELCH SCI	13.76	
	06-15-77	J701405	CHEM STORES	91.93	
	06-22-77	J701441	CHEM STORES	208.75	
	06-22-77	J701441	CHEM STORES	38.87	
	06-22-77	J701442	XEROX REVOLVING CH E	3.60	
	06-23-77	J701474	SERVICE SHOP	8.00	
	06-20-77	7410329	CAHN INSTRUMENTS	423.42	1096.43
	06-01-77	7409265	VWR SCIENTIFIC INC	3053.95	423.42
					3053.95

G & C PROJECT FINANCIAL REPORT NO. 789 7/31/77  
 DEPT 6131 BERG DISTILLATE FUELS BEGIN DATE 07-01-75 END DATE 6-19-78

CURRENT MO. TOTAL TO DATE AMT. AUTH.  
 RECEIPTS .00 75501.72 155932.00

EXPENDITURES			ENCUMBRANCES	TOTAL COMMITTED	BUDGET
SALARY AND WAGES	2945.51	40592.67	8233.91	48826.58	68657.
BENEFITS		1549.78		1549.78	4255.
REP & MAINT	44.42	475.84		475.84	
TRAVEL		1349.29		1349.29	3000.
COMMUNICATIONS		278.32		278.32	
COMPUTER					3000.
SUPPLIES	337.95	13089.19		13089.19	12750.
SUBCONTRACT		150.97		150.97	
EQUIPMENT		8473.11		8473.11	12700.
AWARDS		1450.15		1450.15	6000.
INDIRECT CHARGES	2015.02	28829.64	16740.36	45570.00	45570.
TOTAL	5342.90	96238.96	24974.27	121213.23	155932.
BALANCE	CASH -20737.24		FREE BALANCE	34718.77	

DETAIL TRANSACTION	DATE	NUMBER	DESCRIPTION	AMOUNT	TOTAL
	- -7		TILLERY JAMES ROSS	271.59	
	- -7		HASS GARY R	100.00	
	- -7		HENTON LEE M	400.00	
	- -7		KUJAWA STEPHAN T	400.00	
	- -7		ANDERSON MARK D	400.00	
	- -7		HUSC SILAS A	257.25	
	- -7		MCCANDLESS FRANK P	1116.67	
					2945.51
	07-11-77	8400170	VWR SCIENTIFIC	61.29	
	07-12-77	8400201	APPL SCI LABS INC	28.79	
	07-25-77	8400587	VWR SCIENTIFIC INC	79.64	
	07-20-77	J800007	CHEMISTRY STORES	102.20	
	07-25-77	J800020	CHEM STORES	66.03	
					337.95
	07-12-77	8400203	CAHN INSTRUMENTS	44.42	
ENCUMBRANCE	07-31-70		SALARY ENCUMBRANCE	1300.00	44.42

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G & C

PROJECT FINANCIAL REPORT

NO. 789

7 31 77

DETAIL ENCUMBRANCE	DATE	NUMBER	DESCRIPTION	AMOUNT	TOTAL
	07-31-70		SALARY ENCUMBRANCE	1116.67	
	07-31-70		SALARY ENCUMBRANCE	5817.24	
					8233.91

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G & C PROJECT FINANCIAL REPORT NO. 789 8/31/77  
 DEPT 6131 BERG DISTILLATE FUELS BEGIN DATE 07-01-75 END DATE 6-19-78

	CURRENT MO.	TOTAL TO DATE	AMT. AUTH.	ENCUMBRANCES	TOTAL COMMITTED	BUDGET
RECEIPTS	14520.18	90021.90	155932.00			
EXPENDITURES						
SALARY AND WAGES	4031.01	44623.68		11638.40	56262.08	68657.
BENEFITS	227.49	1777.27			1777.27	4255.
REP & MAINT		475.84			475.84	
TRAVEL	395.00	1744.29			1744.29	3000.
COMMUNICATIONS		278.32			278.32	
COMPUTER						3000.
SUPPLIES	675.49	13764.68			13764.68	12750.
SUBCONTRACT		150.97			150.97	
EQUIPMENT		8473.11			8473.11	12700.
AWARDS		1450.15			1450.15	5000.
INDIRECT CHARGES	2913.24	31742.88		13827.12	45570.00	45570.
TOTAL	8242.23	104481.19		25465.52	129946.71	155932.
BALANCE	CASH -14459.29			FREE BALANCE	25985.29	

DETAIL TRANSACTION	DATE	NUMBER	DESCRIPTION	AMOUNT	TOTAL
	07-27-77	E003083	TREAS OF US	8214.42	
	08-18-77	E005293	US TREAS 1215991	6305.76	14520.18
	- -7		TILLERY JAMES ROSS	271.59	
	- -7		HASS GARY R	100.00	
	- -7		HENTON LEE M	400.00	
	- -7		CONRAD ELIZABETH A	379.50	
	- -7		KUJAWA STEPHAN T	635.00	
	- -7		HASS DANAE A	471.00	
	- -7		ANDERSON MARK D	400.00	
	- -7		HUSD SILAS A	257.25	
	- -7		MCCANDLESS FRANK P	1116.67	4031.01
	08-05-77	J800048	JULY SOC SECURITY	96.26	
	08-05-77	J800053	JULY UCC	3.29	
	08-05-77	J800056	JULY PERS	31.95	
	08-05-77	J800060	JULY TRS	70.49	

DETAIL TRANSACTION	DATE	NUMBER	DESCRIPTION	AMOUNT	TOTAL
	08-05-77	J800085	JULY MT J A	5.49	
	08-25-77	J800141	JULY GROUP MED INS	20.01	
	08-02-77	8400891	ARTHUR H THOMAS CO	4.07	227.49
	08-02-77	8400892	IDAHO VALVE & FITTING	47.41	
	08-08-77	8401040	TYF	125.97	
	08-12-77	8401214	OWENHOUSE HARDWARE	49.42	
	08-15-77	8401241	MATHESON	16.68	
	08-15-77	8401242	VWR SCIENTIFIC	42.00	
	08-15-77	8401243	IDAHO VALVE & FITTING	10.19	
	08-15-77	8401218	MSU BOOKSTORE	42.69	
	08-17-77	8401283	SERVICE ELECTRIC	3.75	
	08-07-77	J800029	CHEM STORES	16.23	
	08-18-77	8401306	LEE HENTON	14.00	
	08-19-77	8401328	VAN WATERS & ROGERS	106.98	
	08-19-77	8401329	H-R OXYGEN & SUPPLY	181.72	
	08-11-77	J800089	LIBRARY XEROX	.66	
	08-18-77	J800101	STOCKROOM	1.12	
	08-23-77	J800122	XEROX REVOL	12.60	
	08-25-77	8401581	PHILLIP MCCANDLESS	395.00	675.49
ENCUMBRANCE	08-31-77		SALARY ENCUMBRANCE	2715.90	395.00
	08-31-77		SALARY ENCUMBRANCE	6350.00	
	08-31-77		SALARY ENCUMBRANCE	2572.50	
					11638.40

UNIVERSITY-TYPE CONTRACTOR'S RECOMMENDATION FOR  
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(See Instructions on Reverse Side)

1. AEC REPORT NO.  FE-2034-8	2. TITLE  CATALYTIC HYDROGENATION OF COAL-DERIVED LIQUIDS
------------------------------------	---

3. TYPE OF DOCUMENT (Check one):

- a. Scientific and technical report
- b. Conference paper:
  - Title of conference \_\_\_\_\_
  - Date of conference \_\_\_\_\_
  - Exact location of conference \_\_\_\_\_
  - Sponsoring organization \_\_\_\_\_
- c. Other (Specify)

4. RECOMMENDED ANNOUNCEMENT AND DISTRIBUTION (Check one):

- a. AEC's normal announcement and distribution procedures may be followed.
- b. Make available only within AEC and to AEC contractors and other U.S. Government agencies and their contractors.

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

Lloyd Berg, Professor

Organization

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

Signature

*Lloyd Berg*

Date

Sept. 21, 1977

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- a. AEC patent clearance has been granted by responsible AEC patent group.
- b. Report has been sent to responsible AEC patent group for clearance.
- c. Patent clearance not required.