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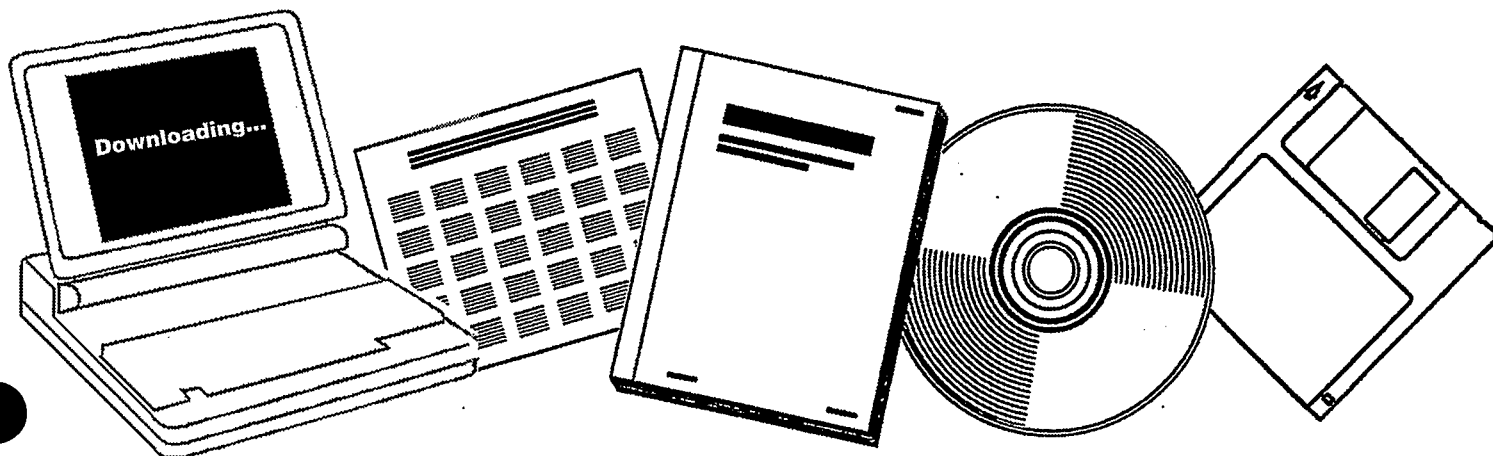
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**IMPROVED CATALYSTS FOR LIQUID HYDROCARBON
FUELS FROM SYNGAS. TECHNICAL PROGRESS
REPORT, APRIL-JUNE 1985**

**UNION CARBIDE CORP., TARRYTOWN, NY.
MOLECULAR SIEVE DEPT**

1985



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TECHNICAL PROGRESS REPORT
DE-AC22-84PC70028

Third Quarterly Report
April - June 1985

IMPROVED CATALYSTS FOR
LIQUID HYDROCARBON FUELS FROM SYNGAS

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Molecular Sieve Department
Catalysts and Process Systems Division

Union Carbide Corporation
Tarrytown Technical Center
Tarrytown, New York 10591

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I. CONTRACT OBJECTIVE

The objective of the contract is to consolidate the advances made during the previous contract in the conversion of syngas to motor fuels using Molecular Sieve-containing catalysts and to demonstrate the practical utility and economic value of the new catalyst/process systems with appropriate laboratory runs.

II. SCHEDULE

The contract work was planned for the twenty-eight month period beginning September 18, 1984.

Work on the program is divided into six tasks.

Task 1 consists of the preparation of a detailed, non-proprietary work plan covering the entire performance of the contract. This work plan was completed in November, 1984.

Task 2 consists of a preliminary techno-economic assessment of the UCC catalyst/process system. This assessment, as well as the final techno-economic evaluation planned for Task 6, will be based on a sensitivity analysis that MITRE is expected to conduct on their recently completed economic evaluation of the Union Carbide Corporation (UCC) system.

Task 3 consists of the optimization of the most promising catalysts developed under prior contract DE-AC22-81PC40077 towards goals defined by the MITRE and Task 2 studies. This work will run through the first 24 months of the contract.

Task 4 consists of the optimization of the UCC catalyst system in a manner that will give it the longest possible service life. This work will run through the first 24 months of the contract.

Task 5 consists of the optimization of a UCC process/catalyst

system based upon a tubular reactor with a recycle loop (i.e., the Arge reactor) containing the most promising catalyst developed under the Tasks 3 and 4 studies. This optimal performance will be estimated from a mathematical model of the tubular reactor which incorporates reaction rate constants determined from appropriate Berty reactor runs. This effort will run through the first 24 months of the contract.

Task 6 consists of an economic evaluation of the optimal performance found under Task 5 for the UCC process/catalyst system. This effort will run from the eighteenth through the twenty-fourth month of the contract, and will be based on the anticipated MITRE sensitivity analysis referred to in the description of Task 2.

The final four months of the contract will be devoted exclusively to the writing of the Eighth Quarterly Report and the Final Technical Report.

III. ORGANIZATION

This contract is being carried out by the Catalyst Research and Development Group of the Molecular Sieve Technology Department, Catalysts and Process Systems Division, Union Carbide Corporation, in Tarrytown, New York.

The principal investigator is Dr. Jule A. Rabo.

The program manager is Dr. Albert C. Frost.

IV. SUMMARY OF PROGRESS

A. Task 1

Task 1, a detailing of the work planned for the other tasks in the contract, has been completed.

B. Task 2

Task 2, a preliminary techno-economic assessment of the UCC catalyst/process system, will be based on a sensitivity analysis that MITRE is expected to conduct on their recently completed economic evaluation of the UCC system.

This sensitivity study is expected to graphically show the differential cost (around the base case cost), expressed as differential cents per gallon of motor fuels, for changes in each of the operating parameters of space velocity, catalyst life, methane make, alpha, C₂₅-C₃₀ carbon cutoff, overall conversion, feed H₂:CO ratio, reactor temperature, and reactor pressure.

These differential cost-operating parameter curves will not only strikingly illuminate which of those operating parameters have the greatest effect on product cost (for Task 2), but they will also be used with catalyst performance data and the existing tubular reactor design curves to readily obtain an economic worth for each tested catalyst for any set of envisioned process conditions (for Task 6).

C. Tasks 3 and 4

The major effort of this Quarter was directed towards improving the formulation of Catalyst 11, which had demonstrated a high initial specific activity (Appendix A of the Second Quarterly Report and Appendix B of this Quarterly Report).

Run 26 revealed the temperature sensitivity of a catalyst promoted with X₉ and X₁₀. At 240C this catalyst showed excellent stability with a specific activity of about 3.1. The catalyst, however, was less stable at higher temperatures, i.e., 250 and 260C. A similar catalyst formulated using UCC-113 in place of UCC-103 (Run 27) demonstrated poor stability even at 240C.

Run 32 revealed the effect of using a promising new additive, X₁₁, in this type of catalyst formulation. The catalyst showed good to excellent stability up to temperatures of 260C, significantly reduced methane make, and improved olefin content of the light gas product. Specific activity at 260C was, however, only 2.0, a value that is comparable to the present best catalyst, Run 15 (Appendix A of the Second Quarterly Report and Appendix B of this Quarterly Report).

Other attempts, at varying the metal concentration, calcining procedures, and additives X₄ and X₃, were unsuccessful.

The preliminary test results of these three runs, as well as five other runs reported for this quarter, are listed in Appendix A as Runs 26-33. Additional, detailed analyses of these runs will be presented in the next quarterly report.

Such detailed analyses for the runs (10-25) reported for the

second quarter in a preliminary manner are listed in Appendix B.

D. Task 5

A comparison between the UCC and the Gulf-Badger (as sketchily described in a 1983 Hydrocarbon Processing article) catalyst/process systems indicates (with many assumptions) that the Gulf-Badger system may operate at a 40-50C lower temperature and an ill-defined higher H₂:CO ratio than does the UCC system to give a product distribution having slightly more naphtha but less distillate and wax. See Appendix C for details.

A small error was found in the FIXED computer program used to simulate the ARGE/UCC process design. This error has been corrected, and the revised program was used to check out some of the old design data sent to MITRE for their recently completed economic study. It was found that the required changes to the MITRE study were limited to easily made changes to the stated specific activity, and not to any of the stream compositions or space velocities. Consequently, only minor corrections will have to be made to the MITRE study.

E. Task 6

Since this final techno-economic evaluation is scheduled to begin in Fiscal Year 1986, no work was done on it this quarter.

Additionally, the sequential sensitivity studies expected from MITRE will substantially aid in satisfying the objectives of this task in addition to completing those of Task 2 (see B. Task 2).

V. CHANGES

There were no contract changes during the Third Quarter.

VI. FUTURE WORK

Task 2 will be deferred until the expected MITRE sensitivity study is completed.

Tasks 3 and 4 will continue to be devoted to developing new, stable catalyst formulations that will have higher specific activities and lower methane makes than do our present catalysts.

Task 5 will be devoted to incorporating heat generation and heat transfer terms into the presently isothermal mathematical model, so that upper space velocity limits can be defined for different operating pressures.



A. C. Frost

APPENDIX A. CATALYST TESTING: SUMMARY OF RUNS
REPORTED DURING THIS QUARTER

APPENDIX A. CATALYST TESTING: SUMMARY OF RUNS
REPORTED DURING THIS QUARTER

J. G. Miller, L. F. Elek, C-L Yang and P. K. Coughlin

This report is organized around the eight catalytic tests conducted from April through June 1985, the third quarter of this contract.

A list of the catalysts tested and descriptions of their preparations are shown in Table A1. All of the catalysts tested involved cobalt oxide intimately contacted with UCC-103, except for Run 27 in which UCC-113 was substituted for UCC-103 as the catalyst support. One of the eight catalysts, Run 33, was prepared by the method developed in the previous three year contract (DE-AC22-81PC40077), while the remainder were prepared by the method used for the catalyst tested in Run 11 (Appendix A of the Second Quarterly Report and Appendix B of this report) of the present contract.

An abbreviated table of results for these catalyst runs is shown in Table A2. The conversion, weight percent CH_4 , weight percent C_5^+ , specific activity, the methane factor and a qualitative estimate of stability are listed for each catalyst. A more complete report of results and analyses for these runs will be presented in the Fourth Quarterly Report.

Table A1. Description of most of the catalysts tested during the third quarter.

Run Catalyst	Catalyst preparation
26 Co/X ₉ /X ₁₀ /UCC-103 (12185-14)	The X ₉ and X ₁₀ promoted cobalt oxide was formed in close contact with UCC-103 by the method used in Run 11 (similar to Run 20). The resulting powder was bonded with 15% silica and extruded to 1/8" pellets. Theoretical pct Co=11.9, pct X ₉ =0.5, pct X ₁₀ =0.7.
27 Co/X ₉ /X ₁₀ /UCC-113 (12200-15)	The X ₉ and X ₁₀ promoted cobalt oxide catalyst was formulated by the method used for Catalyst 26, except that UCC-103 was replaced by UCC-113 (same formulation as Run 24). Theoretical pct Co=7.9, pct X ₉ =0.37, pct X ₁₀ =0.50.
28 Co/X ₉ /X ₁₀ /X ₄ /UCC-103 (12200-17)	The X ₉ and X ₁₀ promoted cobalt oxide catalyst was formulated similarly to Run 26, then further promoted with X ₄ . Theoretical pct Co=4.1, pct X ₉ =0.19, pct X ₁₀ =0.25, pct X ₄ =0.58.
29 Co/X ₉ /X ₁₀ /UCC-103 (12200-18)	The X ₉ and X ₁₀ promoted cobalt oxide catalyst was formulated similarly to Run 26. Theoretical pct Co=7.8, pct X ₉ =0.35, pct X ₁₀ =0.47.
30 Co/X ₉ /X ₁₀ /X ₃ /UCC-103 (12185-16)	The X ₉ , X ₁₀ , X ₃ promoted cobalt oxide catalyst was formulated similarly to Run 26. Theoretical pct Co=7.8, pct X ₉ =0.35, pct X ₁₀ =0.47, pct X ₃ =0.06.
31 Co/X ₉ /X ₁₀ /UCC-103 (12185-17)	The X ₉ and X ₁₀ promoted cobalt oxide catalyst was prepared using the same formulation as that used in Run 29, except that the preparation contained no calcination steps. Theoretical pct Co=7.8, pct X ₉ =0.35, pct X ₁₀ =0.47.
32 Co/X ₁₁ /UCC-103 (12200-19)	The X ₁₁ promoted cobalt oxide catalyst was formulated similarly to Run 26. Theoretical pct Co=8.2, pct X ₁₁ =1.2.
33 Co/X ₉ /X ₁₀ /X ₄ /X ₃ /UCC-103 (12185-18)	The X ₉ , X ₁₀ , X ₃ promoted cobalt oxide was formed in close contact with UCC-103 by the method used in Run 15, then further promoted with X ₄ . The resulting powder was bonded with 15% silica and extruded to 1/8" pellets. Theoretical pct Co=8.2, pct X ₉ =0.37, pct X ₁₀ =0.49, pct X ₄ =0.48, pct X ₃ =0.06.

Table A2. Preliminary catalyst test results for most of the runs made during the third quarter.

Run	Catalyst	Hours on stream	Total conversion (CO+H ₂)	CH ₄ wt %	C ₅ ⁺ wt %	Specific activity	Methane factor(1)	Stability
26	Co/X ₉ /X ₁₀ /UCC-103 (12185-14)	67.5	45.3	8.1	82.7	3.10	1.57	Excellent (2)
		163.0	44.5	7.9	83.0	3.11	1.60	
		186.5	51.3	11.2	78.5	2.75	2.60	Fair (3)
		332.7	50.1	12.2	76.7	2.29	3.08	
27	Co/X ₉ /X ₁₀ /UCC-113 (12200-15)	67.0	40.5	8.02	82.0	2.64	1.91	Fair (2)
		139.5	38.2	8.39	81.6	2.55	2.00	
		188.5	43.7	12.9	75.5	1.88	3.22	Fair (3)
		306.0	42.0	13.4	73.7	1.67	4.32	
28	Co/X ₉ /X ₁₀ /X ₄ /UCC-103 (12200-17)	44.0	27.0	18.2	66.5	1.04	3.22	— (2)
		68.0	26.4	14.8	73.4	0.97	2.89	
29	Co/X ₉ /X ₁₀ /UCC-103 (12200-18)	49.5	45.1	9.4	82.5	2.11	2.97	Fair (3)
		146.0	38.0	11.2	80.0	1.49	3.64	
30	Co/X ₉ /X ₁₀ /X ₃ /UCC-103 (12185-16)	114.0	48.7	8.3	81.2	3.09	1.99	Fair (3)
		258.0	41.7	10.8	77.3	2.08	3.10	
31	Co/X ₉ /X ₁₀ /UCC-103 (12185-17)	22.5	27.2	20.8	59.4	0.93	3.43	Poor (2)
		94.0	22.1	19.2	64.8	0.62	3.12	
32	Co/X ₁₁ /UCC-103 (12200-19)	43.5	46.5	5.43	85.8	3.11	1.03	Excellent (2)
		187.0	42.1	5.31	84.5	2.55	0.96	
		211.0	56.5	7.95	81.1	2.11	1.68	Very Good (4)
		403.0	57.6	8.63	80.8	1.86	1.79	
		427.0	62.9	14.2	71.9	1.55	2.85	
499.0	55.7	17.7	66.7	1.31	3.32	Poor (5)		
33	Co/X ₉ /X ₁₀ /X ₄ /X ₃ /UCC-103 (12185-18)	44.5	59.7	12.2	73.0	2.48	2.04	Poor (4)
		116.5	52.0	15.5	69.7	1.30	2.79	

(1) The ratio of the amount of CH₄ actually produced to the amount of CH₄ predicted from the Schulz-Flory equation, $[\text{CH}_4/(1-\alpha)^2]$.

(2) Conditions 240C, 300 psig, 1:1 H₂:CO, 300 GHSV.

(3) " 250C " " " " " " "

(4) " 260C " " " " " " "

(5) " 270C " " " " " " "

Appendix B. CATALYST TESTING: DETAILS OF RUNS
REPORTED DURING LAST QUARTER

Appendix B. CATALYST TESTING: DETAILS OF RUNS
REPORTED DURING LAST QUARTER

J. G. Miller, L. F. Elek, C-L Yang and P. K. Coughlin

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I. INTRODUCTION

This report presents detailed analyses of the sixteen catalyst test runs summarized in Appendix A of the Second Quarterly Report, which constituted the major thrust of the work during that quarter.

All sixteen catalysts contained either cobalt oxide or iron oxide, in each case intimately mixed with a Molecular Sieve. Fifteen of them also contained the shape selective component UCC-103; one contained a newly developed shape selective component, UCC-113.

Two different methods of preparation were employed, and the catalysts prepared by each method were used to explore several lines of investigation.

With seven of the catalysts, prepared by the same method employed in the previous contract (DE-AC22-81PC40077), the following possibilities were investigated:

1. Whether performance can be improved by raising the metal loading.
2. Reproducing the results obtained in the previous contract.
3. The effects of replacing radioactive thoria with X_4 and X_{10} .
4. The effectiveness of the additives UCC-101 and UCC-112.
5. Whether a spent catalyst can be regenerated with hydrogen.

Preparation of the nine remaining catalysts involved a method of intimately contacting a Fischer-Tropsch active metal with UCC-103 or UCC-113, which has been newly developed with the objective of improving the catalyst's activity and stability. These were used to explore:

1. The effectiveness in these catalysts of three additives-- X₉, X₁₀ and X₄--which were found effective in catalysts prepared by the previous method.
2. The effect of replacing the Molecular Sieve UCC-103 with the newly developed UCC-113.
3. The effectiveness of a catalyst based on potassium-promoted iron.

II. Run 10 (12185-06) with Catalyst 10 (Co/Th/X₄/UCC-103)

This run continued the search for improvements on the performance of the most promising catalyst to date (Catalyst 6, Run 11677-11, Third Annual Report, Contract DE-AC22-81PC40077). Specifically, the purpose was to test a substantially higher level of cobalt promoted with thorium and X₄.

The thorium-promoted cobalt oxide was formed in close contact with UCC-103 and further promoted with X₄. The resulting powder was bonded with 15 percent silica, then extruded to 1/8-inch pellets. The final catalyst contained theoretically 18.8 percent cobalt, 2.9 percent thorium and 1.7 percent X₄. Catalyst 11677-11, in contrast, contained 4.5 percent cobalt. Catalyst 4, Run 12185-03, of the First Quarterly Report contained nearly as much cobalt (17.0 percent) as this one, but no thorium or X₄.

Conversion, product selectivity, isomerization of the pentane, and percent olefins of the C₄'s are plotted against time on stream in Figs. B1-4. Simulated distillations of the C₅⁺ product are plotted in Figs. B5-12. Carbon number product distributions are plotted in Figs. B13-20. Chromatograms from simulated distillations are reproduced in Figs. B21-28. Detailed material balances appear in Tables B1-2.

The specific activity, initially about 2.5, fell off steeply to about 1.3 at 116 hours on stream; assuming this catalyst had

the same specific activity per percent cobalt as Catalyst 11677-11, it would be predicted to have a specific activity on the order of 3.8. Thus, although this catalyst contained four times the cobalt concentration of 11677-11, and approximately the same concentration as Catalyst 12185-03 with the addition of thorium and X₄, it was substantially less active than either. The initial specific activity of Catalyst 12185-03, for example, was approximately 8 (but with poor stability).

Following its initial deactivation, the catalyst appeared very stable for the remaining 70 hours of the run, but this was too short a period to permit reliable conclusions.

Production of methane was significantly lower than with the two other catalysts. Following are the ratios of weight percent methane experimentally observed, to weight percent predicted by the mathematical model:

12185-06 Co/Th/X ₄ /UCC-103	0.6:1
11677-11 Co/Th/X ₄ /UCC-103+UCC-101	1.0:1
12185-03 Co/UCC-103	1.2:1

The calculated Schulz-Flory alpha value was fairly high at 0.86, corresponding to a high wax production of about 12 percent. The Schulz-Flory plots show a fairly linear product distribution with little or no observed carbon number cut-off.

This catalyst's use of cobalt was not very efficient. Its selectivity for C₅⁺, however, was very good, and following the initial deactivation period its stability was, at least potentially, very good as well.

RUN A2185-06

111 H₂CO
300 Psig
860°C

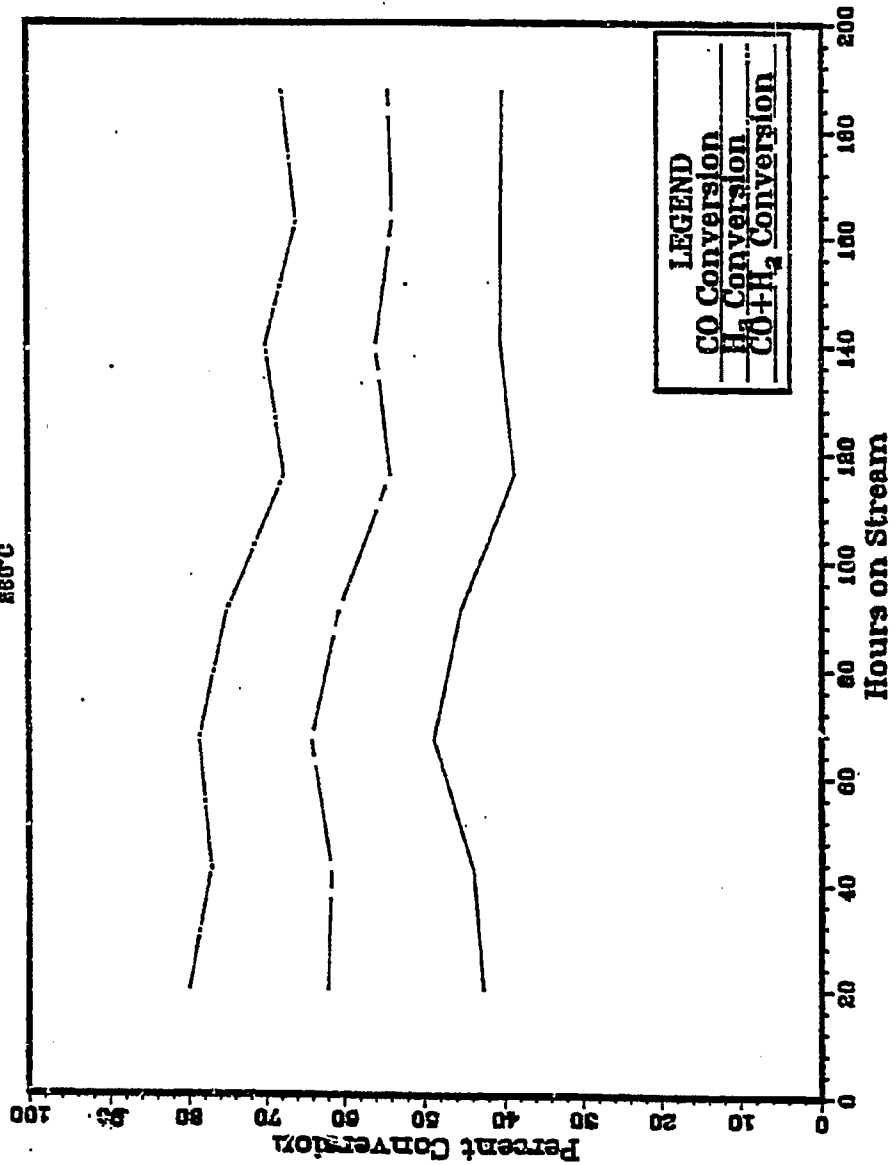


Fig. B1

RUN 12185-06

111 H₂CO
300 PSIG
280°C

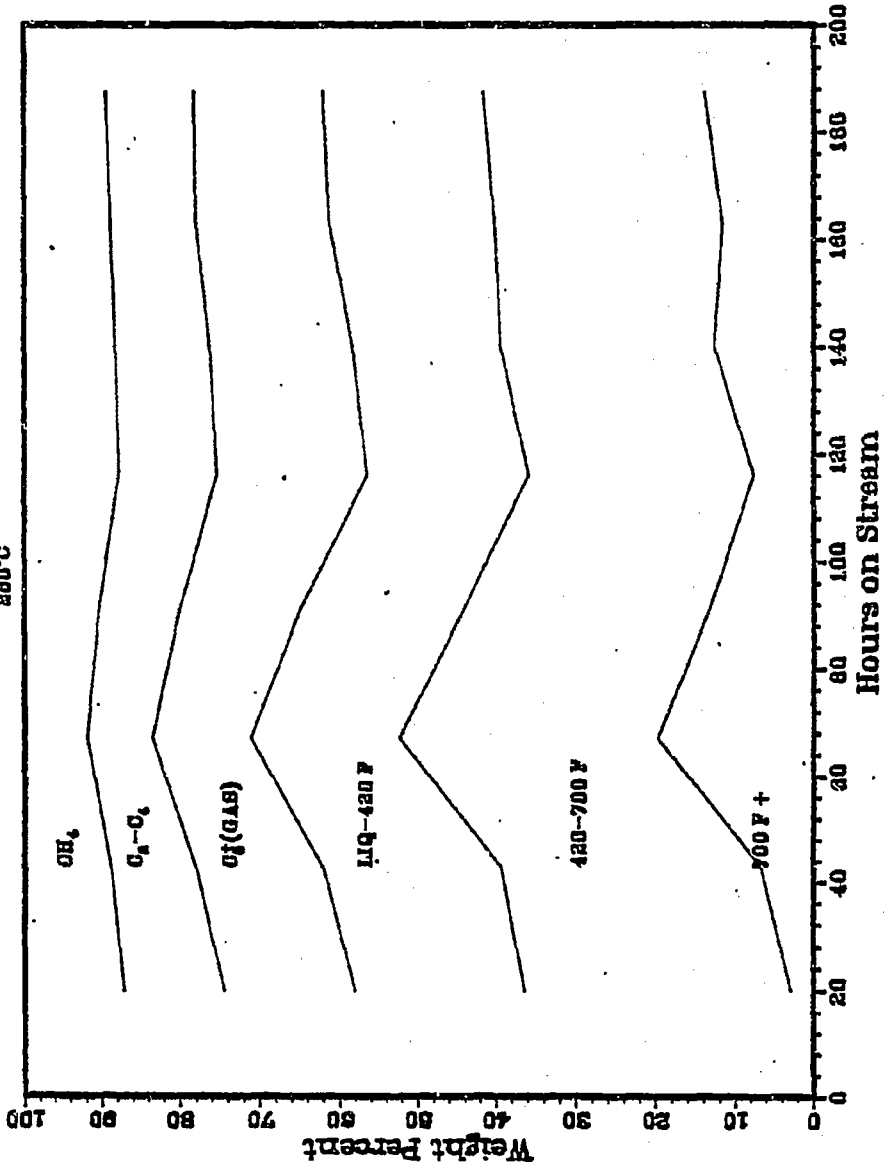


Fig. B2

RUN 12185-06

111 H₂O
300 PSIG
260°C

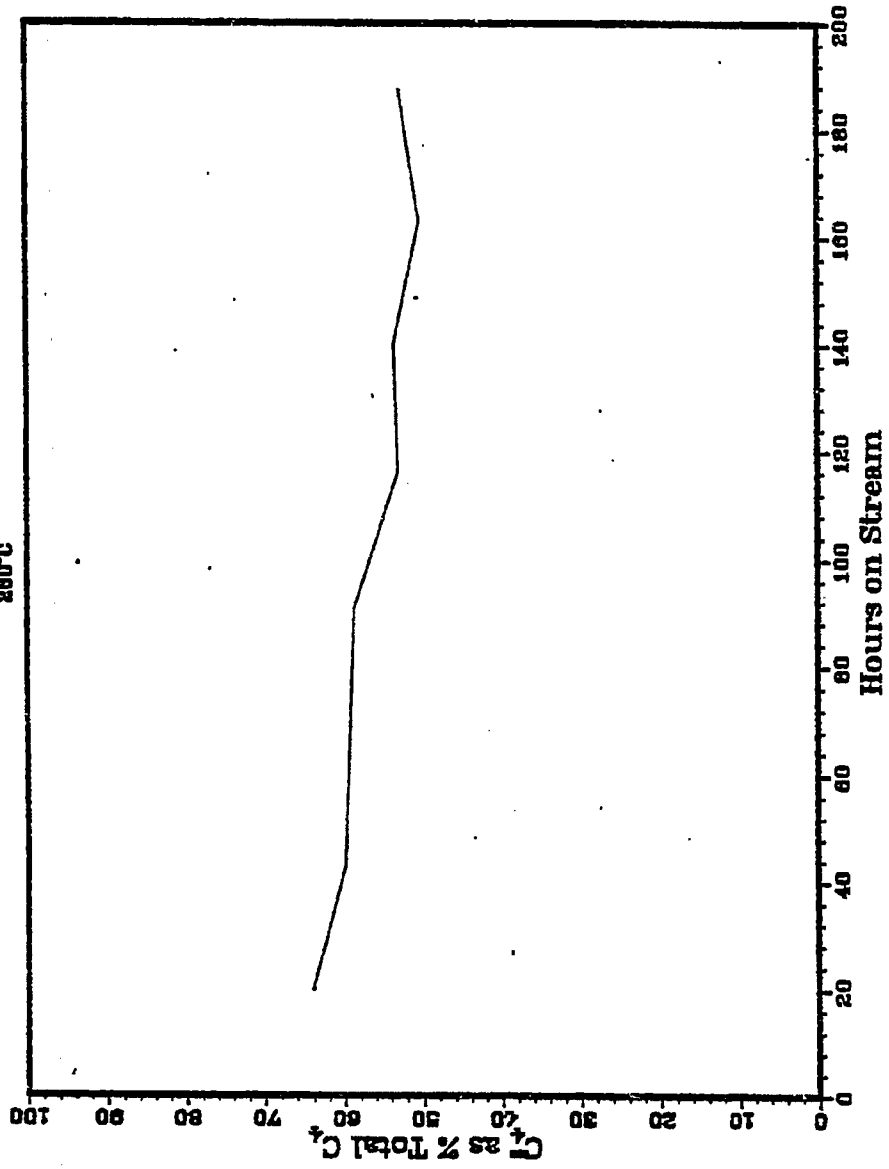


Fig. B3

RUN 12185-06

111 H₂O
300 PSIG
260°C

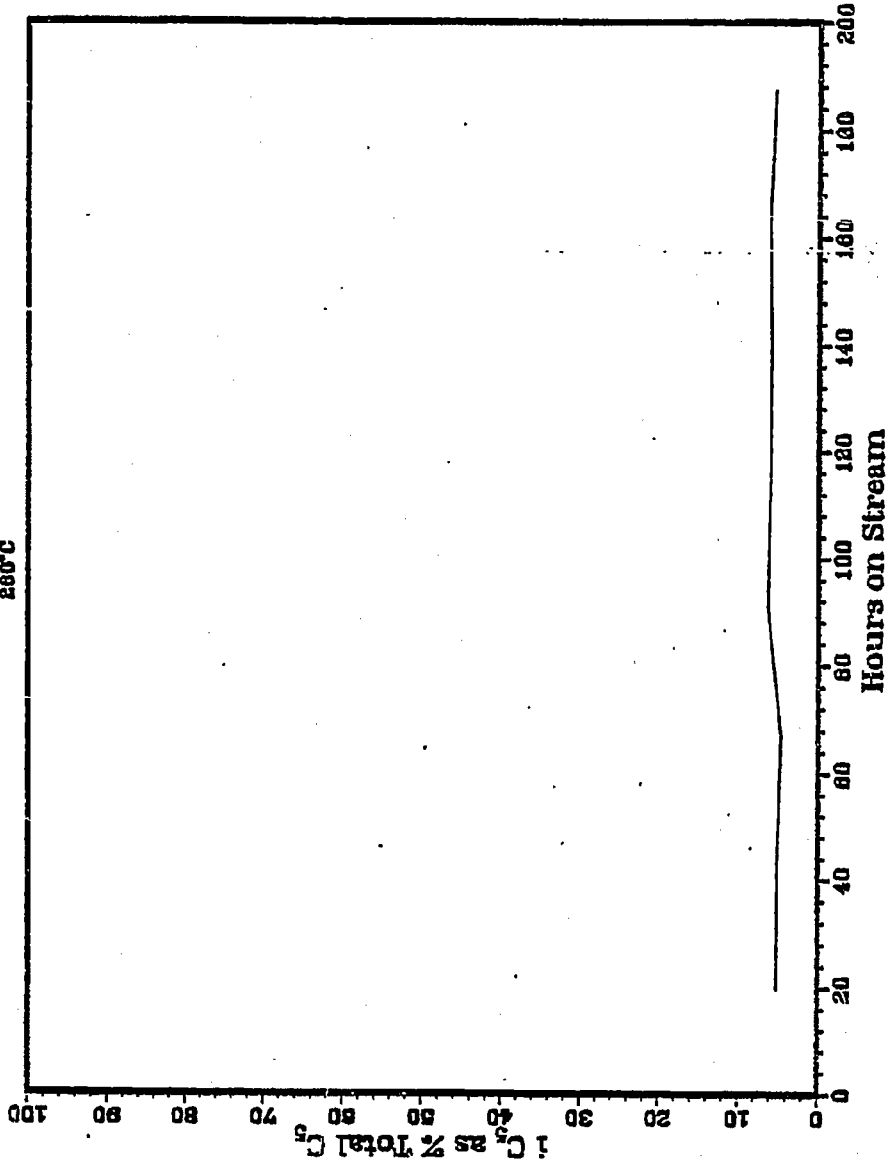


Fig. B4

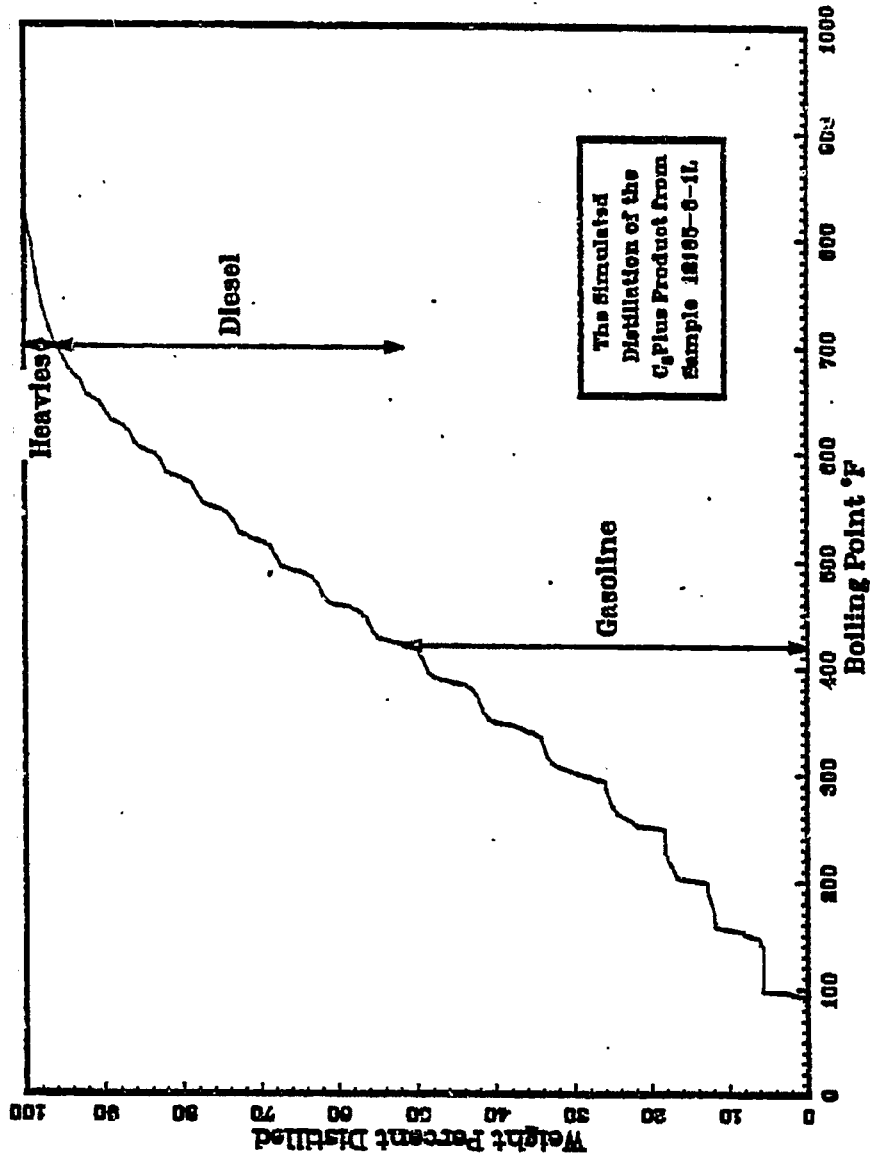
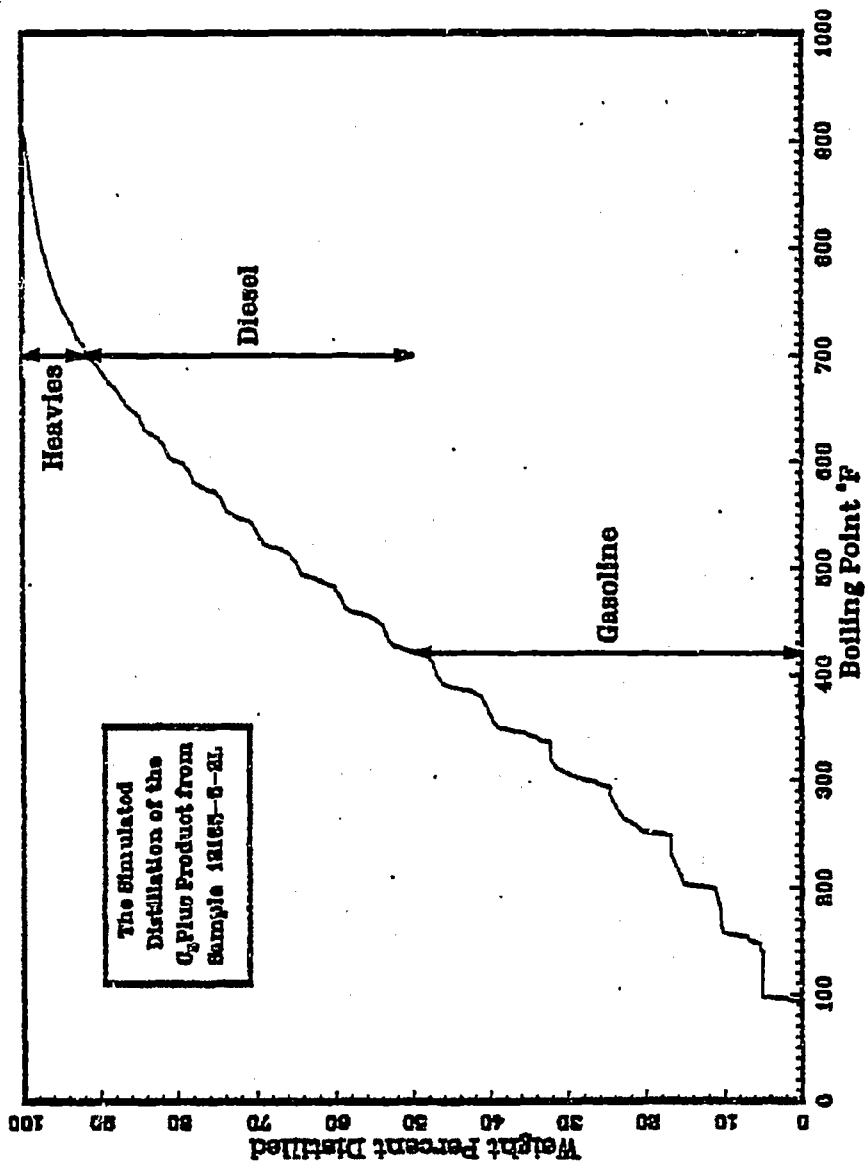


Fig. B5



The Simulated
Distillation of the
C₃Plus Product from
Sample 12165-C-2L

Fig. B6

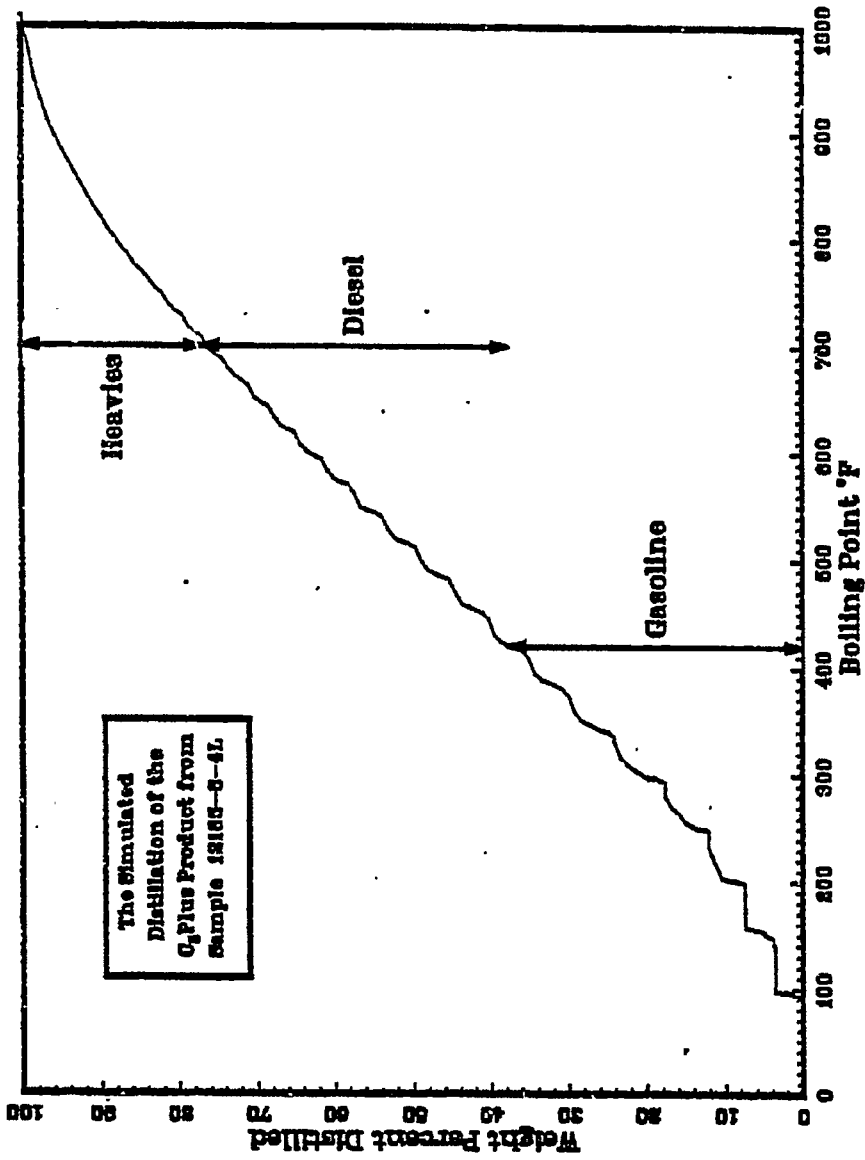
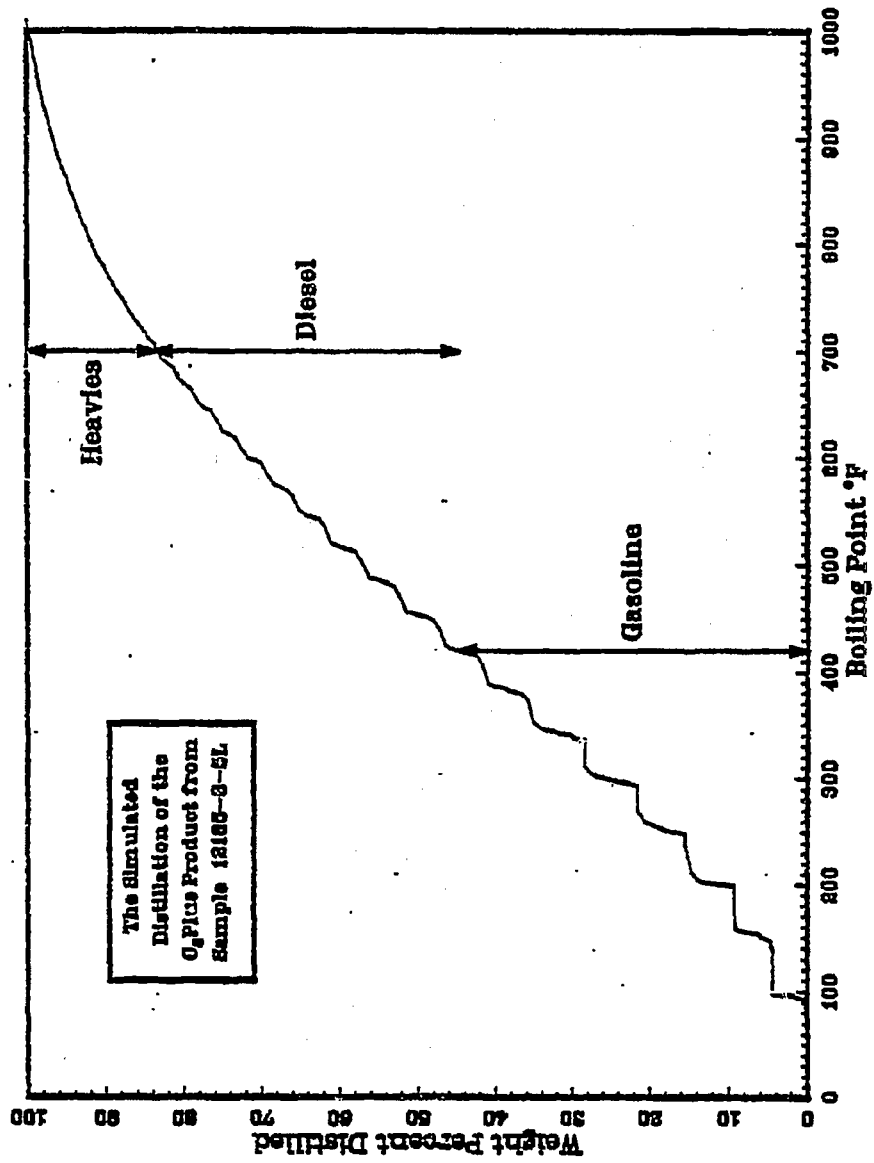
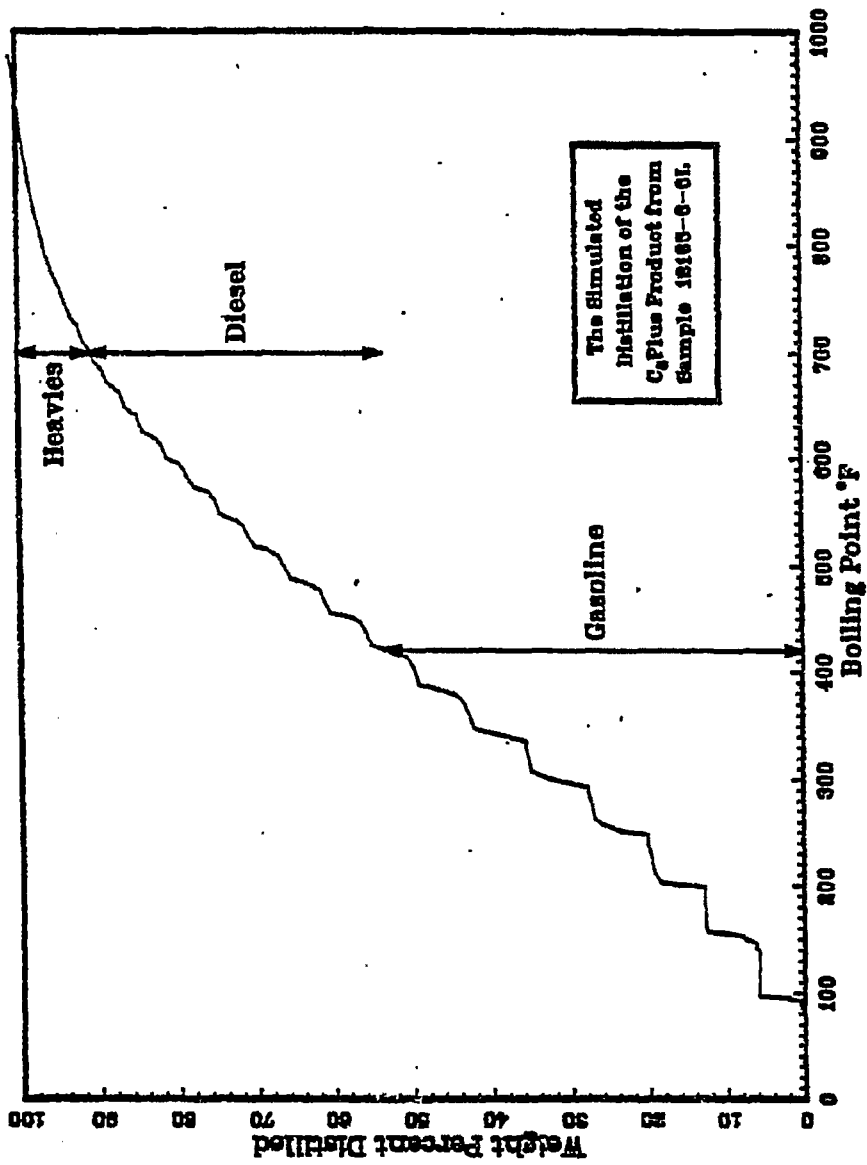


Fig. B7



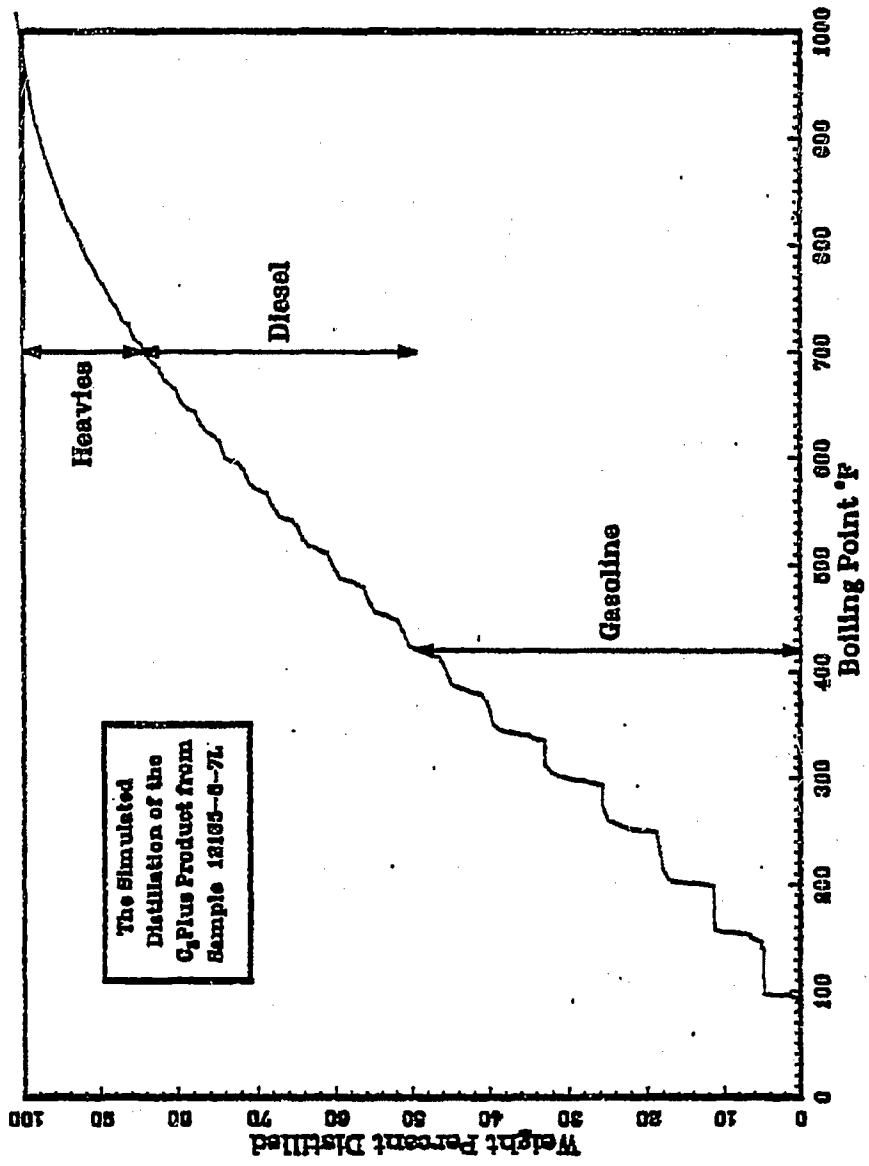
The Simulated
Distillation of the
O.Pica Product from
Sample 12185-3-5L

Fig. B8



The Simulated
Distillation of the
C₈ Plus Product from
Sample 18185-8-01.

Fig. B9



The Simulated
Distillation of the
C₁+ Plus Product from
Sample 12165-0-7L

Fig. B10

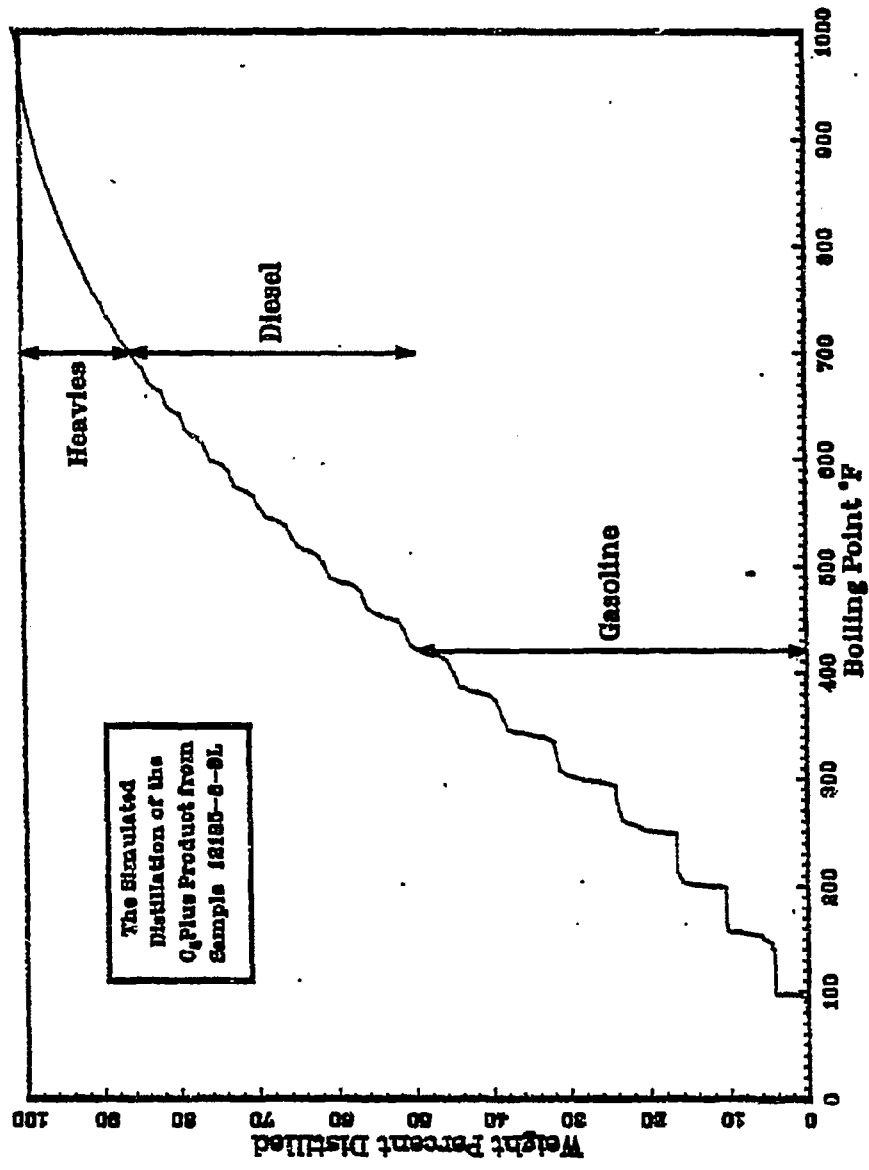


Fig. B11

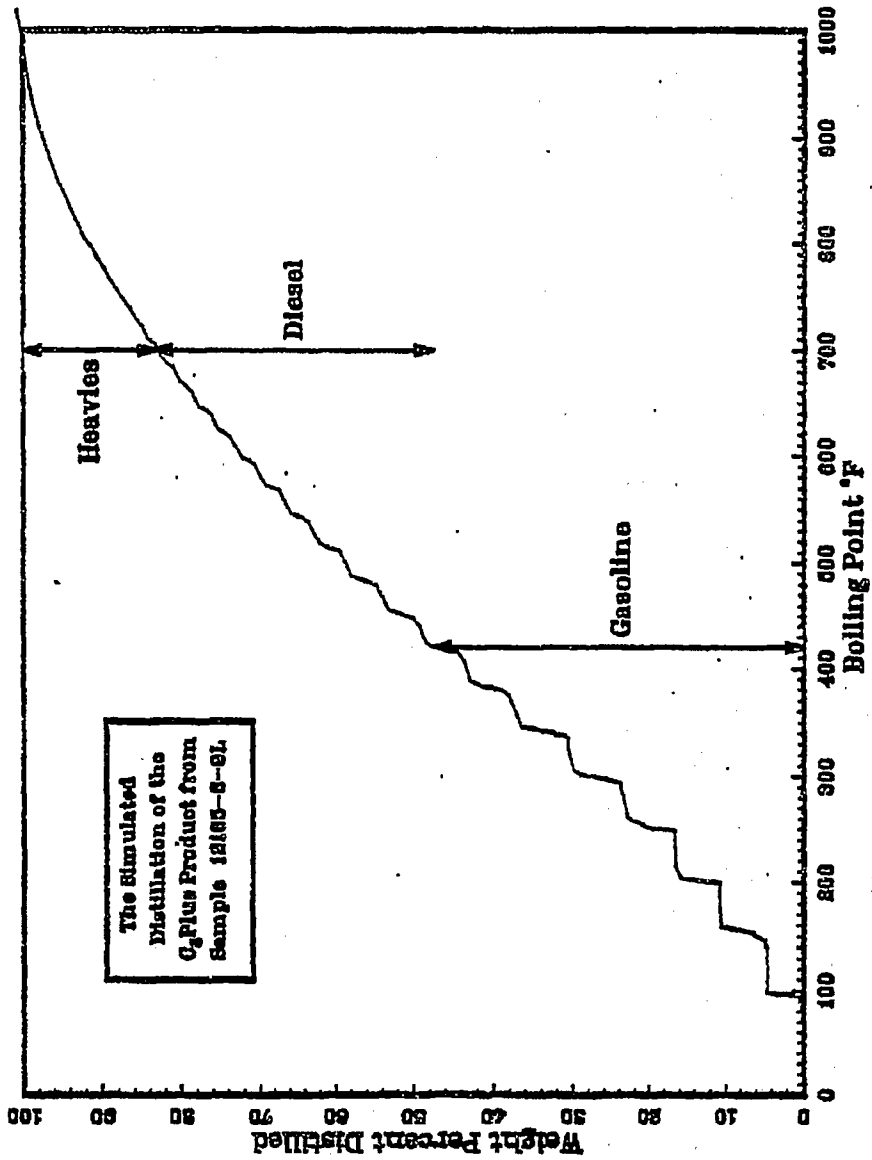


Fig. B12

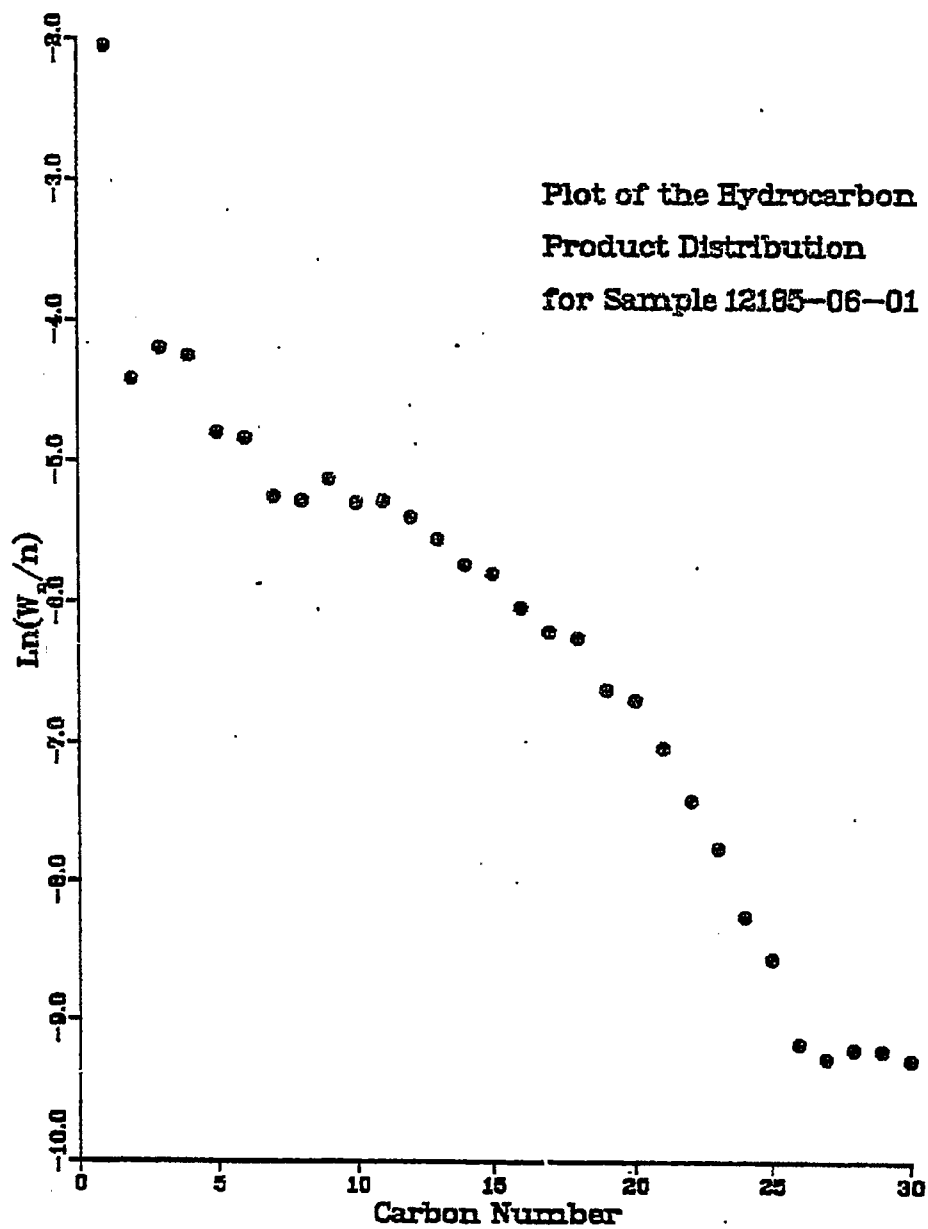


Fig. B13

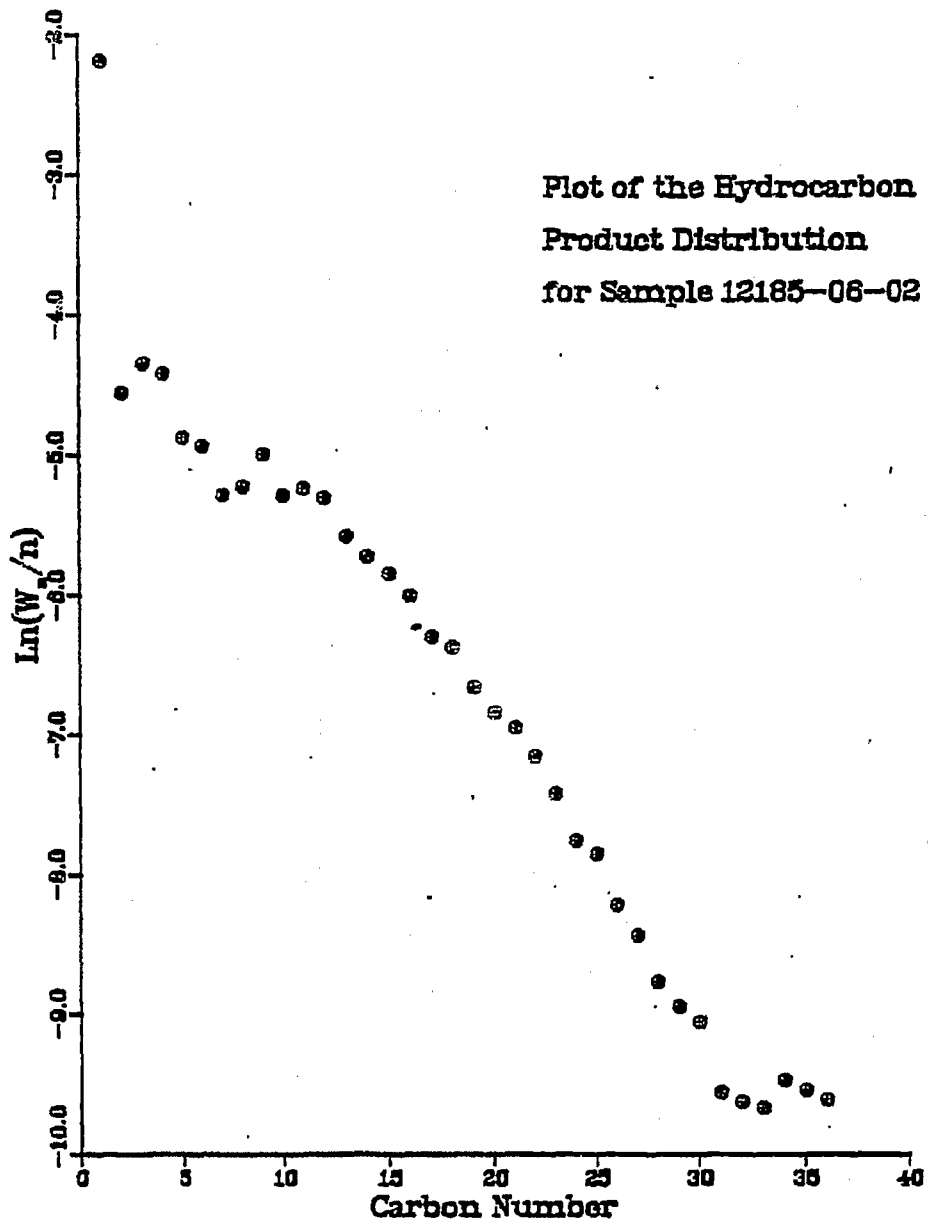


Fig. B14

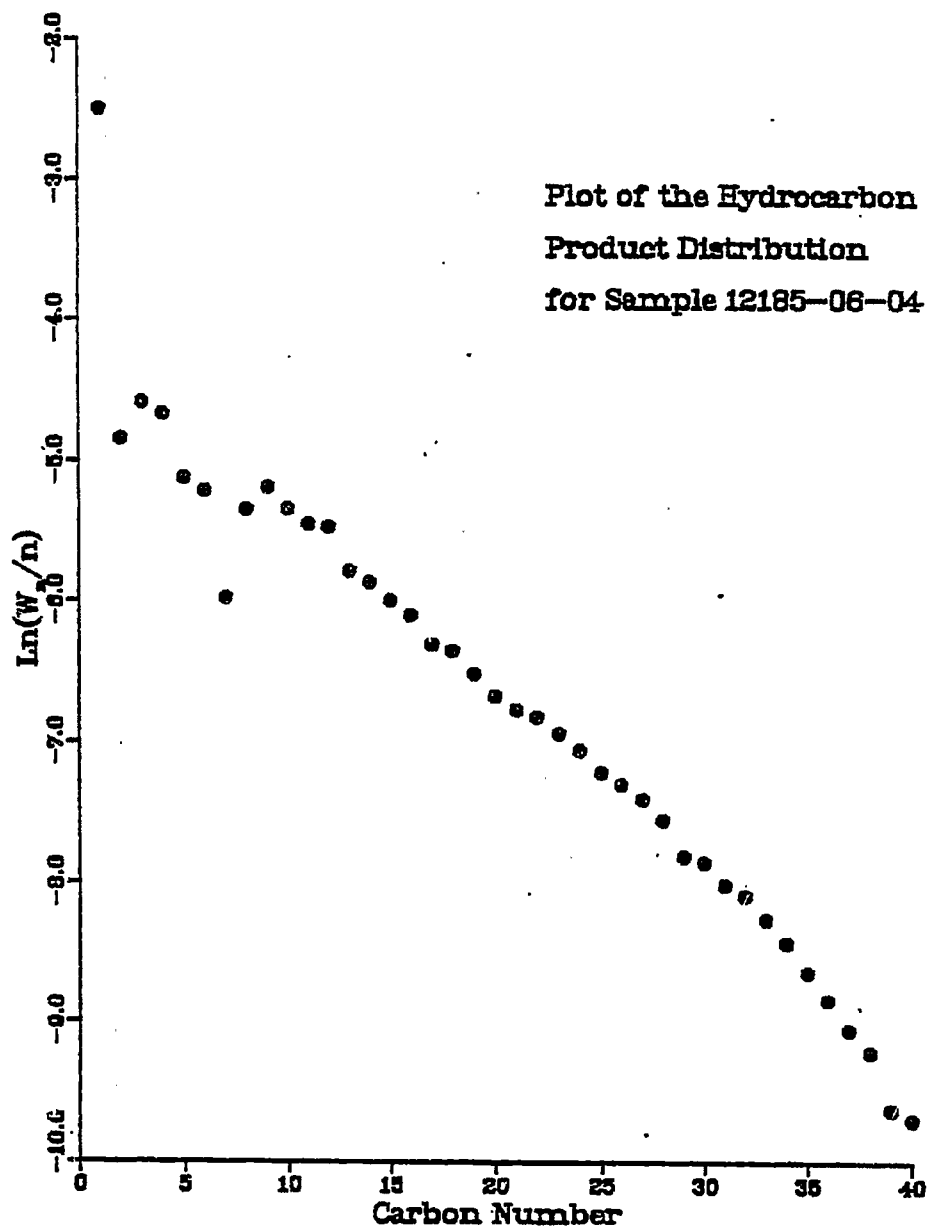


Fig. B15

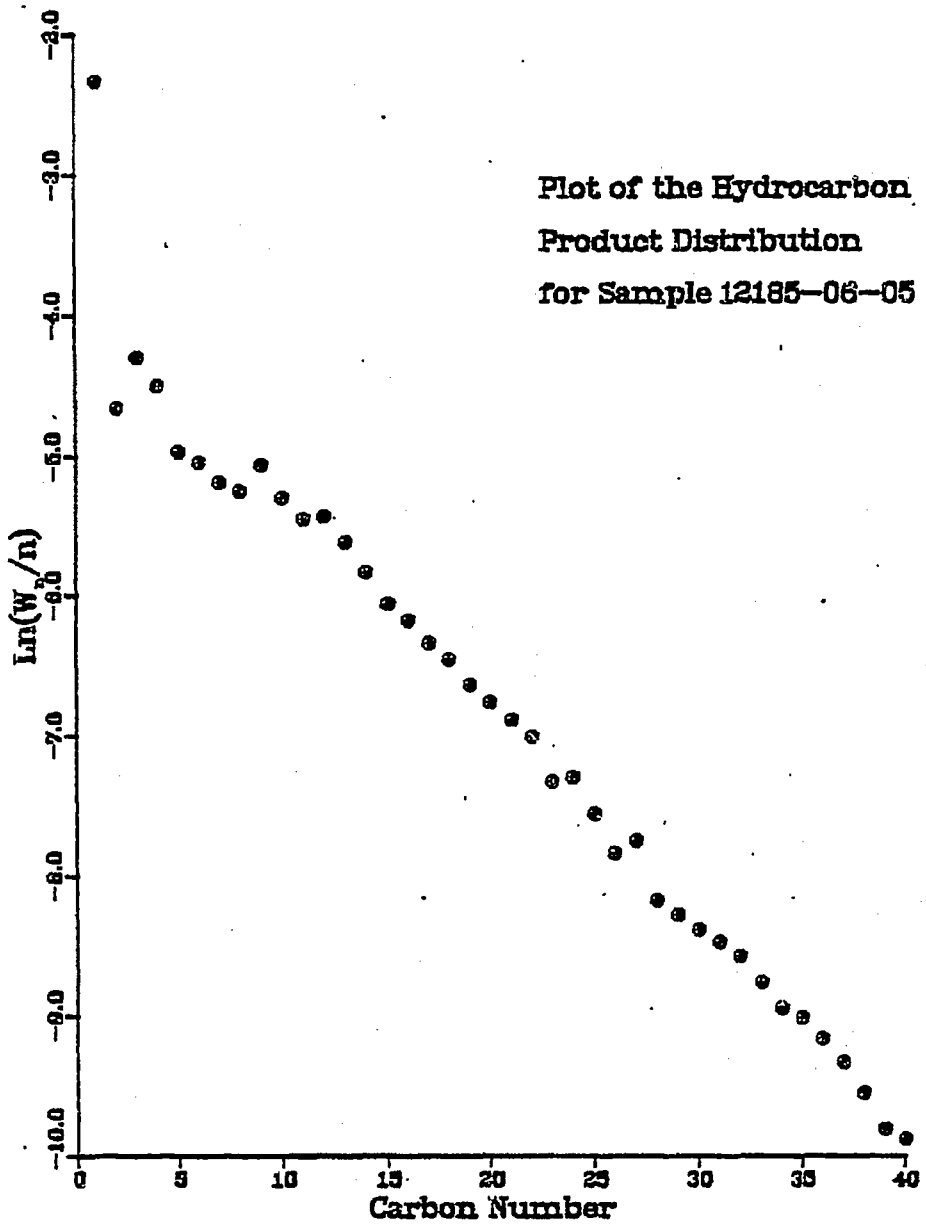


Fig. B16

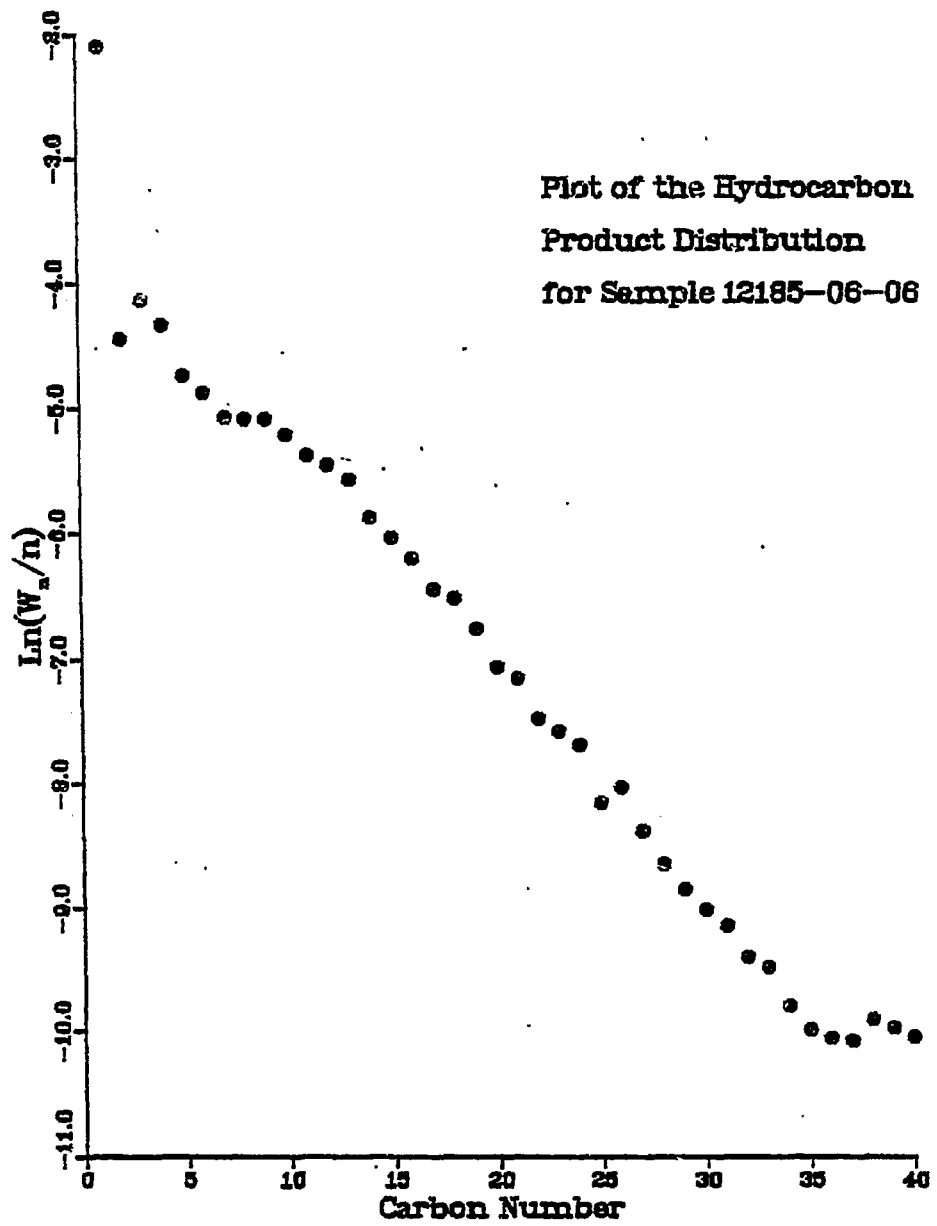


Fig. B17

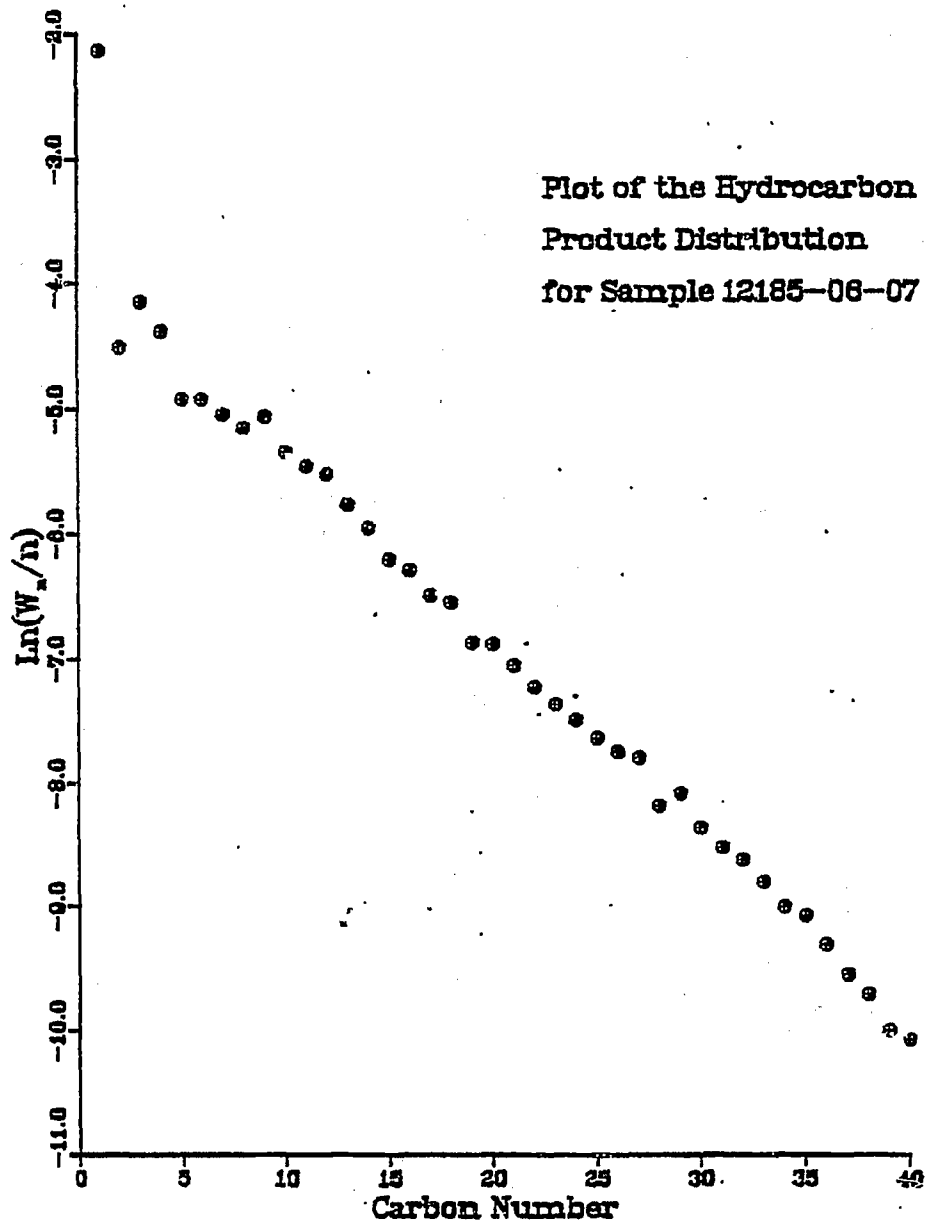


Fig. B18

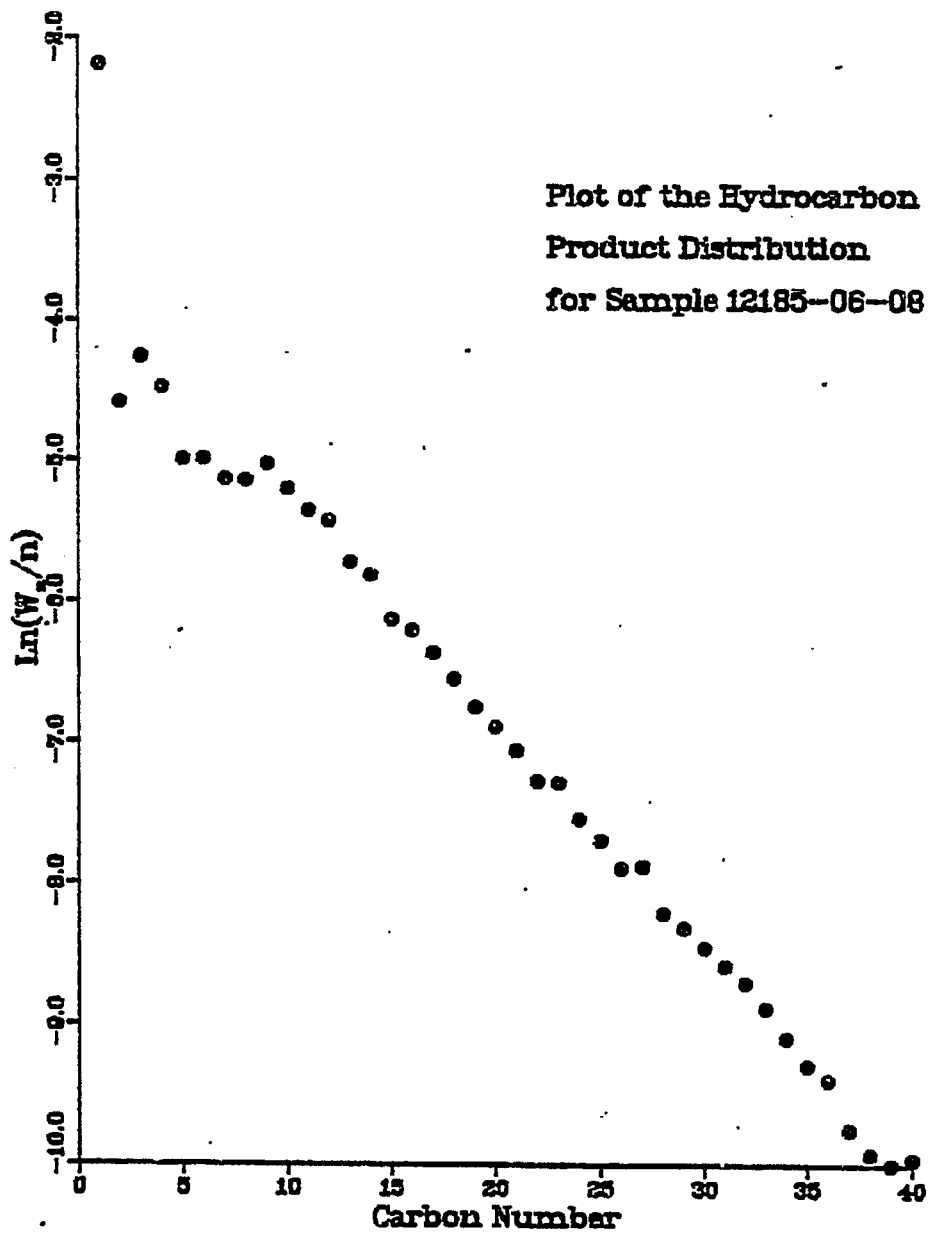


Fig. B19

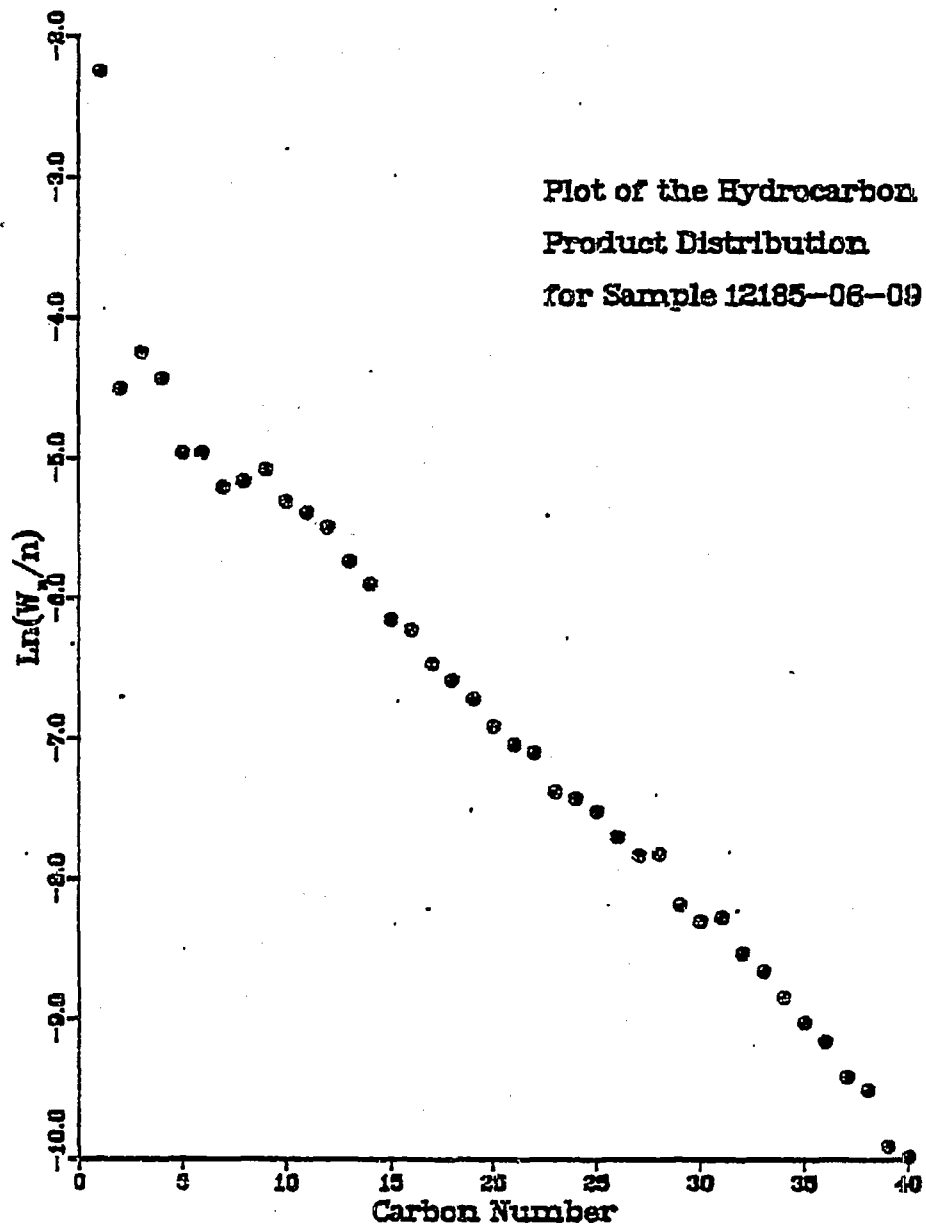
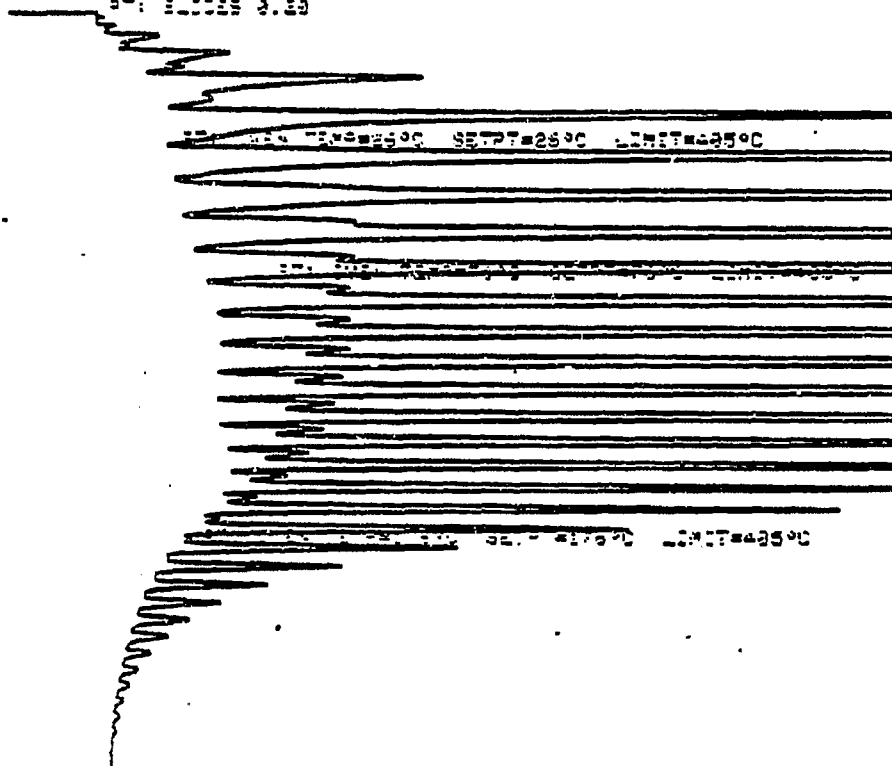


Fig. B20

OVER TEMP NOT PERM

ST: 11000 3.80



TEMP=2500 SETPT=2500 LIMIT=2500

TEMP=2500 SETPT=2500 LIMIT=2500

TEMP=2500 SETPT=2500 LIMIT=2500

ST: 11000 TEMP=2700 SETPT=2700 LIMIT=4050

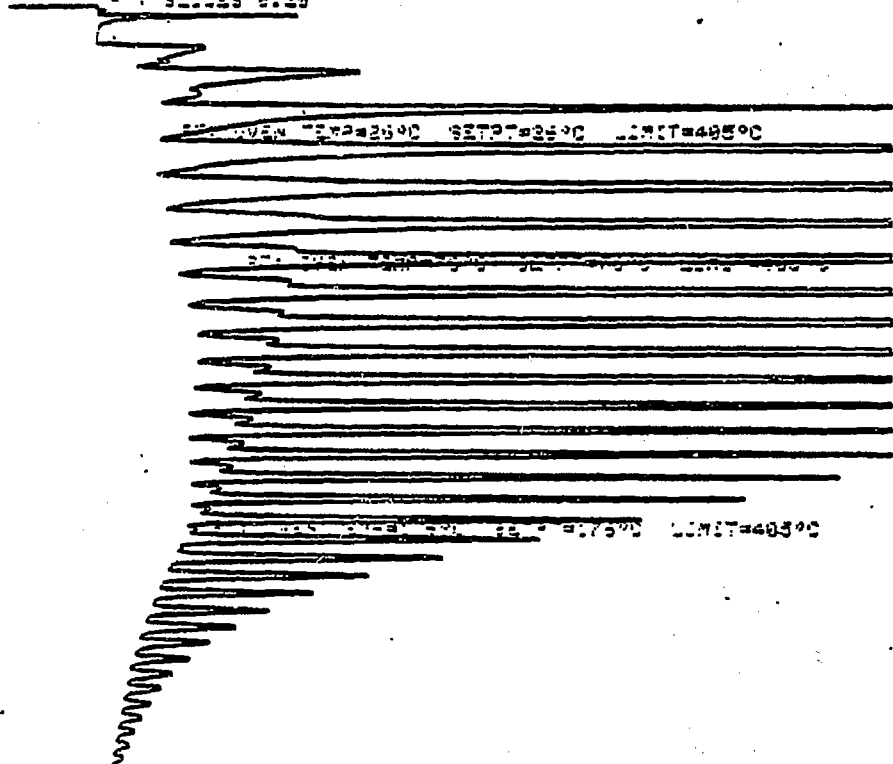
ST: 11000 TEMP=3500 SETPT=3500 LIMIT=4050

ST: 11000

12185-06-01
Fig. B21

IVEN TEPD NOT TEPD

001 5.1225 0.20



IVEN TEPD=2600 SETPT=2500 LIMIT=4050C

0050=LIMIT

001 IVEN TEPD=2750 SETPT=2750 LIMIT=4050C

001 IVEN TEPD=3300 SETPT=3300 LIMIT=4050C

12135-06-02
Fig. B22

OPEN TEMP CONTROL

SET POINT 26.00

OPEN TEMP=26.00 SETPT=26.00 LIMIT=40.00

OPEN TEMP=26.00 SETPT=26.00 LIMIT=40.00

OPEN TEMP=26.00 SETPT=26.00 LIMIT=40.00

OPEN TEMP=27.00 SETPT=27.00 LIMIT=40.00

OPEN TEMP=35.00 SETPT=35.00 LIMIT=40.00

END

12185-06-04

12185-06-04

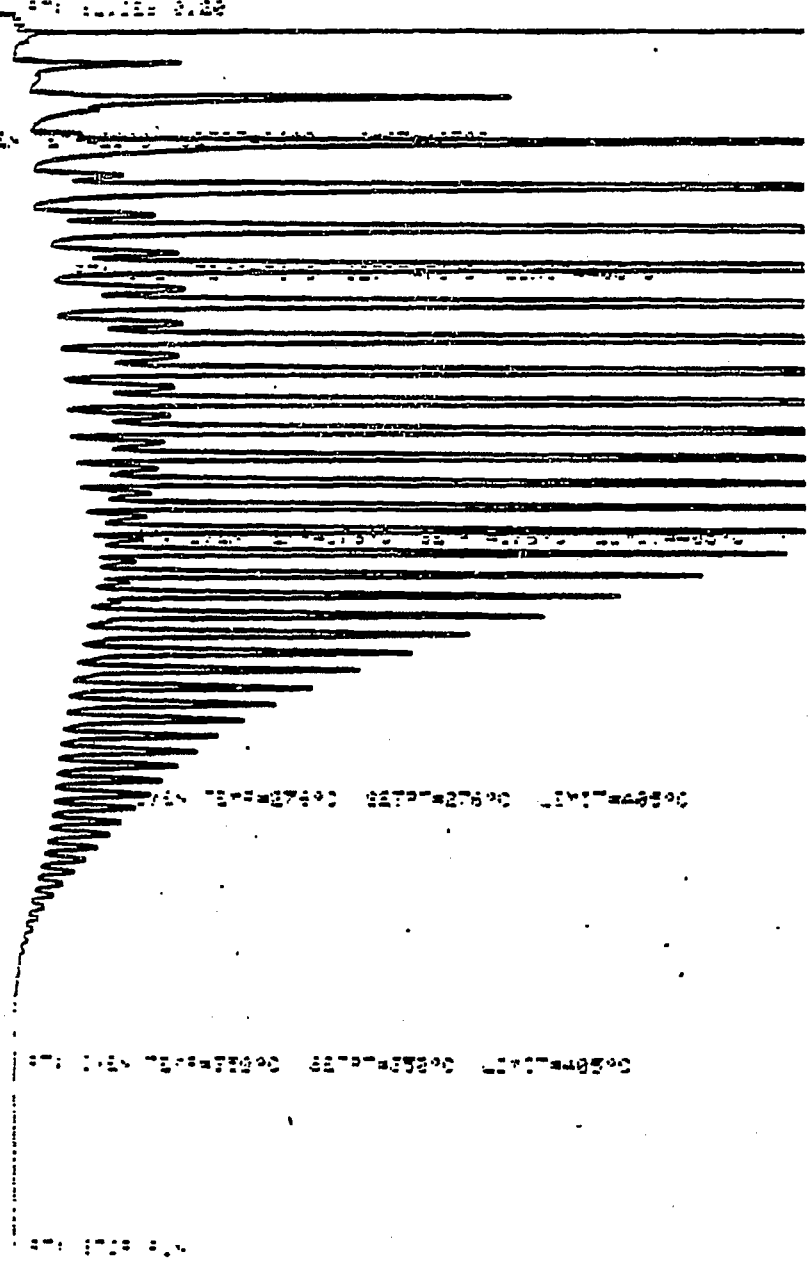
Fig. B23

001

OVEN TEMP AT 2500

001 11:11 0.00

001 OVEN



12135-06-05

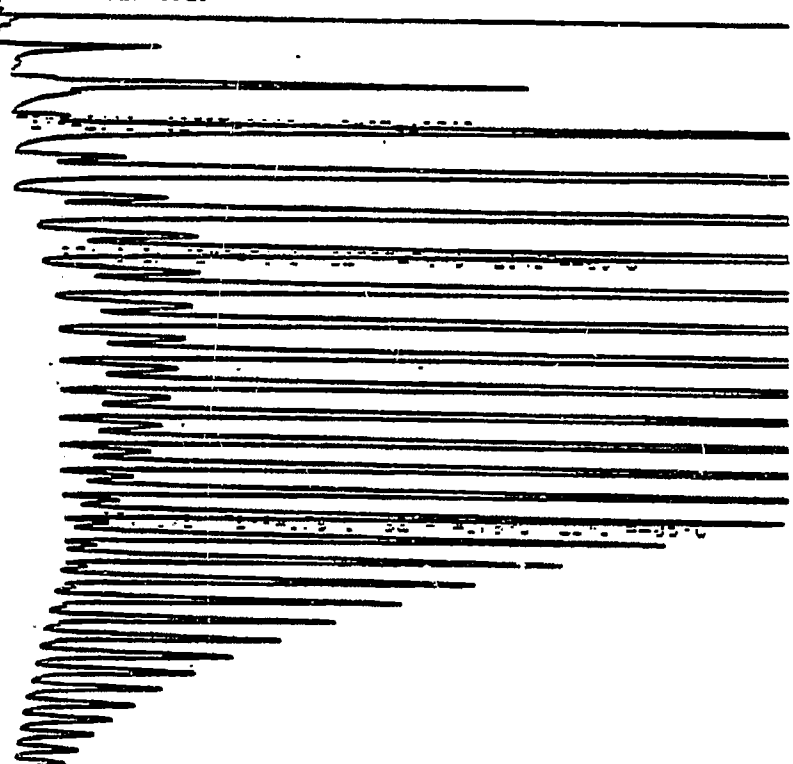
Fig. B24

DATA TIME NOT RECORDED

TOT

ST: 0.0000 0.00

ST: 0.0000



ST: 0.0000 0.0000 0.0000

ST: 0.0000 0.0000 0.0000

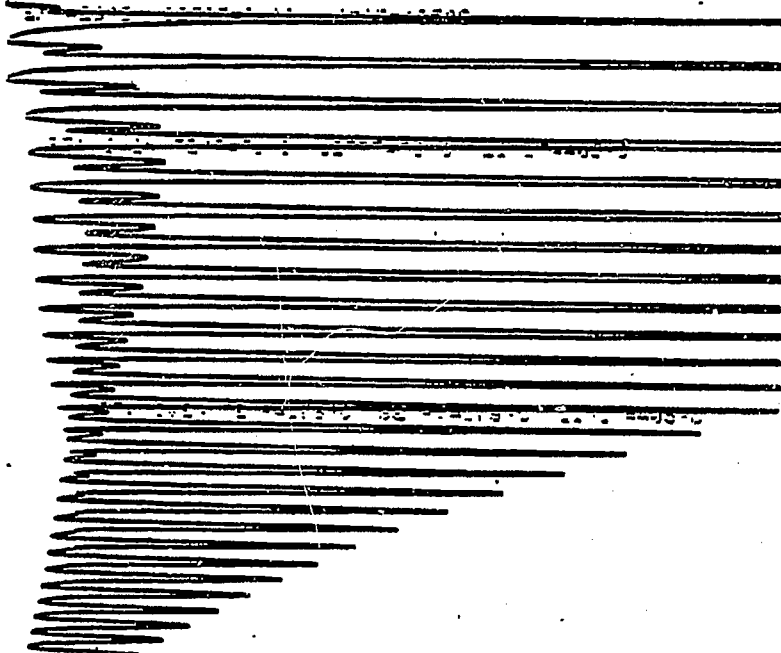
12185-06-06

Fig. B25

DATA FROM 1950

DATE: 11-1-50

DATA FROM 1950



DATA FROM 1950 SECRETARY LIMIT 4000

DATA FROM 1950 SECRETARY LIMIT 4000

12185-06-07

Fig. B26

0-24 750 107 880

0-1 1111 0.20

0-1 0-1

0-1 1-1 1111 0.20

0-1 1-1 1111 0.20

0-1 1-1

12:05-06:08

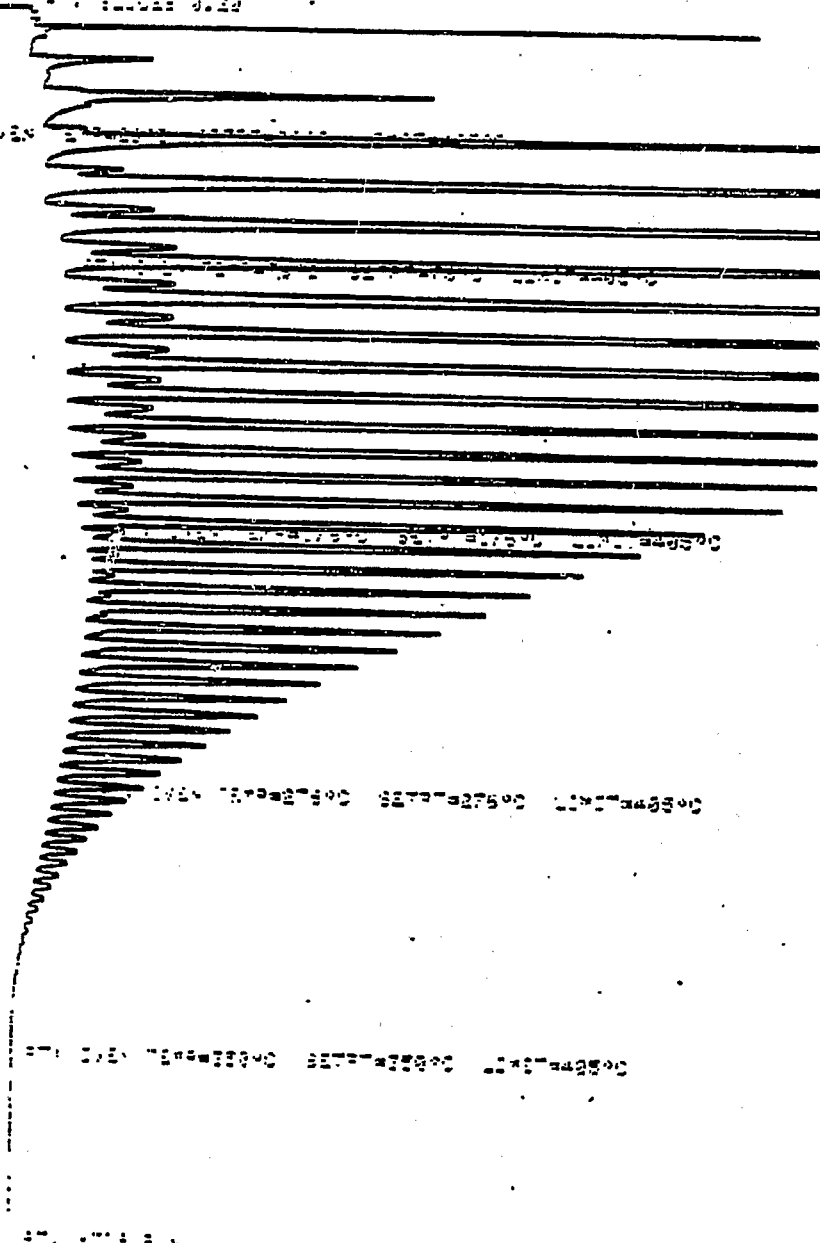
Fig. B27

OPEN FILE NO. 1000

DATE: 11/22/55

NO. 1000

100



12185-06-09

Fig. B28

RESULT OF SYNGAS OPERATION

RUN NO. 12185-06
 CATALYST CO/TH/X4-U103 12006-48 80 CC 36.76 GM (49.76 AFTER RUN +13.G)
 FEED H2:CO OF 50:50 @400CC/MN OR 300 GHSV

RUN & SAMPLE NO.	12185-06-01	185-06-02	185-06-04	185-06-05	185-06-06
FEED H2:CO:AR	50:50: 0	50:50: 0	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	20.0	43.0	67.0	91.0	116.0
PRESSURE, PSIG	300	300	300	300	300
TEMP. C	261	261	260	260	260
FEED CC/MIN	400	400	400	400	400
HOURS FEEDING	20.00	23.00	20.00	24.00	25.00
EFFLNT GAS LITER	178.00	219.75	205.65	258.50	297.64
GM AQUEOUS LAYER	61.73	72.14	56.72	62.11	67.19
GM OIL	21.04	30.36	43.50	42.86	30.69
MATERIAL BALANCE					
GM ATOM CARBON %	76.50	81.50	99.29	95.72	89.34
GM ATOM HYDROGEN %	82.99	93.20	105.35	100.85	100.93
GM ATOM OXYGEN %	94.35	94.16	94.73	92.31	94.90
RATIO CHX/(H2O+CO2)	0.5879	0.7027	1.1179	1.0963	0.8463
RATIO X IN CHX	2.3705	2.3391	2.2829	2.3118	2.3668
USAGE H2/CO PRODT	2.0283	2.0064	1.7109	1.7419	1.9720
FEED H2/CO FRM EFFLNT	1.0849	1.1436	1.0610	1.0536	1.1297
RESIDUAL H2/CO RATIO	0.3812	0.4682	0.4417	0.4816	0.5965
RATIO CO2/(H2O+CO2)	0.1666	0.1370	0.1341	0.1304	0.1119
K SHIFT IN EFFLNT	0.0762	0.0743	0.0684	0.0722	0.0752
SPECIFIC ACTIVITY SA	2.5673	1.9621	2.6727	2.1241	1.2304
CONVERSION					
ON CO %	42.72	43.91	48.79	45.39	38.76
ON H2 %	79.88	77.03	78.68	75.04	67.67
ON CO+H2 %	62.06	61.58	64.18	60.60	54.10
PRDT SELECTIVITY, WT %					
CH4	12.89	11.16	8.23	9.67	12.30
C2 HC'S	2.42	2.10	1.58	1.90	2.34
C3H8	1.91	1.84	1.44	1.90	2.63
C3H6=	2.63	2.03	1.61	2.18	2.20
C4H10	2.10	1.96	1.57	1.87	2.52
C4H8=	3.62	2.82	2.19	2.56	2.73
C5H12	2.85	2.65	2.10	2.43	3.10
C5H10=	1.28	1.17	0.88	1.05	1.29
C6H14	3.03	2.92	2.22	2.60	3.31
C6H12= & CYCLO'S	1.72	1.37	1.03	1.22	1.26
C7+ IN GAS	7.47	7.85	6.01	7.68	9.85
LIQ HC'S	58.09	62.13	71.14	64.95	56.46
TOTAL	100.00	100.00	100.00	100.00	100.00

Table B1

SUB-GROUPING					
C1 -CA	25.57	21.92	16.62	20.07	24.73
CS -420 F	38.01	38.63	31.09	35.76	39.42
420-700 F	33.52	32.74	32.51	30.85	28.28
700-END PT	2.90	6.71	19.78	13.32	7.57
CS+-END PT	74.43	78.08	83.38	79.93	75.27
ISO/NORMAL MOLE RATIO					
C4	0.0181	0.0181	0.0185	0.0167	0.0173
C5	0.0531	0.0527	0.0478	0.0664	0.0631
C6	0.0962	0.0897	0.0721	0.0718	0.0810
C4=	0.0488	0.0569	0.0551	0.0619	0.0686
PARAFFIN/OLEFIN RATIO					
C3	0.6921	0.8685	0.8528	0.8318	1.1378
C4	0.5614	0.6710	0.6900	0.7058	0.8894
C5	2.1656	2.2007	2.3274	2.2575	2.3285
SCHULZ-FLORY DISTRBTM					
ALPHA (EXP(SLOPE))	0.8281	0.8523	0.8973	0.8703	0.8453
RATIO CHA/(1-A)**2	4.3612	5.1204	7.7949	5.7419	5.1385
LIQ HC COLLECTION					
PHYS. APPEARANCE	GLD OIL	OIL WAX	OIL WAX	OIL WAX	OIL WAX
DENSITY (* 40 C)	0.7410	0.6970	0.7730	0.7500	0.7690
N, REFRACTIVE INDEX	1.4260	1.423*	1.430*	1.425*	1.421*
SIMULT'D DISTILATN					
10 WT % @ DEG F	299	299	310	301	300
16	316	320	351	342	339
50	461	483	563	516	484
84	625	661	786	737	673
90	655	710	839	802	732
RANGE(16-84 %)	309	341	435	395	334
WT % @ 420 F	37.30	36.50	26.50	32.00	36.50
WT % @ 700 F	95.00	89.20	72.20	79.50	86.60

Table B1, cont

RESULT OF SYNGAS OPERATION

RUN NO. 12185-06
 CATALYST CO/TH/X4-U103 12006-48 80 CC 36.76 GM (49.76 AFTER RUN +13.G)
 FEED H2:CO OF 50:50 @400 CC/MN OR 300 GHSV

RUN & SAMPLE NO.	12185-06-07	185-06-08	185-06-09
FEED H2:CO:AR	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	140.0	163.0	187.5
PRESSURE, PSIG	300	300	300
TEMP. C	261	260	260
FEED CC/MIN	400	400	400
HOURS FEEDING	24.00	23.00	24.50
EFFLNT GAS LITER	285.49	286.37	311.72
GM AQUEOUS LAYER	64.81	54.01	54.49
GM OIL	33.69	34.54	40.13
MATERIAL BALANCE			
GM ATOM CARBON %	93.95	94.84	101.63
GM ATOM HYDROGEN %	102.99	104.32	103.69
GM ATOM OXYGEN %	96.59	92.51	95.21
RATIO CHX/(H2O+CO2)	0.9277	1.0724	1.2088
RATIO X IN CHX	2.3575	2.3471	2.3365
USAGE H2/CO PRDCT	1.8944	1.7971	1.7218
FEED H2/CO FRM EFFLNT	1.0962	1.1000	1.0202
RESIDUAL H2/CO RATIO	0.5523	0.6273	0.5475
RATIO CO2/(H2O+CO2)	0.1161	0.1184	0.1216
K SHIFT IN EFFLNT	0.0725	0.0843	0.0758
SPECIFIC ACTIVITY SA	1.3883	1.2202	1.4934
CONVERSION			
ON CO %	40.52	40.41	40.26
ON H2 %	70.03	66.02	67.94
ON CO+H2 %	55.96	53.82	54.24
PRDCT SELECTIVITY, WT %			
CH4	11.80	11.22	10.60
C2 HC'S	2.21	2.04	2.20
C3H8	2.55	2.51	2.36
C3H6=	2.19	1.73	1.93
C4H10	2.38	2.30	2.27
C4H8=	2.63	2.24	2.44
C5H12	3.02	2.87	2.93
C5H10=	0.62	0.51	0.55
C6H14	3.19	3.07	3.06
C6H12= & CYCLO'S	1.20	1.01	1.14
C7+ IN GAS	9.82	9.19	8.41
LIQ HC'S	58.39	61.30	62.11
TOTAL	100.00	100.00	100.00

Table B2

SUB-GROUPING			
C1 -C4	23.75	22.05	21.80
C5 -420 F	36.83	37.80	36.58
420-700 F	26.74	28.50	27.70
700-END PT	12.67	11.65	13.91
C5+-END PT	76.25	77.95	78.20
ISO/NORMAL MOLE RATIO			
C4	0.0160	0.0197	0.0174
C5	0.0615	0.0643	0.0569
C6	0.0754	0.0872	0.0677
C4=	0.0687	0.0751	0.0672
PARAFFIN/OLEFIN RATIO			
C3	1.1116	1.3819	1.1650
C4	0.8719	0.9891	0.8986
C5	4.7111	5.4209	5.1809
SCHULZ-FLORY DISTRBTN			
ALPHA (EXP(SLOPE))	0.8623	0.8628	0.8684
RATIO CH4/(1-A)**2	6.2255	5.9602	6.1189
LIQ HC COLLECTION			
PHYS. APPEARANCE	OIL WAX	OIL WAX	OIL WAX
DENSITY (* 40 C)	0.7620	0.7710	0.7750
N, REFRACTIVE INDEX	1.4245*	1.4235*	1.4245*
SIMULT'D DISTILATN			
10 WT % @ DEG F	301	301	301
16	342	342	342
50	516	504	515
84	749	727	756
90	813	788	818
RANGE(16-84 %)	407	385	414
WT % @ 420 F	32.50	34.50	33.00
WT % @ 700 F	78.30	81.00	77.60

Table B2, cont

III. Run 11 (12200-06) with Catalyst 11 (Co/UCC-103)

The purpose of this run was to test a new method of combining cobalt oxide in close contact with UCC-103, intended to yield a more active Fischer-Tropsch catalyst. The cobalt oxide was formed in close contact with UCC-103 using the new procedure. The resulting powder was bonded with 15 weight percent silica and extruded to 1/8-inch pellets. The final catalyst contained 12.8 weight percent cobalt.

Conversion, product selectivity, isomerization of the pentane, and percent olefins of the C₄'s are plotted against time on stream in Figs. B29-32. Simulated distillations of the C₅⁺ product are plotted in Figs. B33-39. Carbon number product distributions are plotted in Figs. B40-46. Chromatograms from simulated distillations are reproduced in Figs. B47-53. Detailed material balances appear in Tables B3-4.

This catalyst demonstrated some unusual properties not observed in any previous catalyst.

Most significant of these was a remarkably high initial syn-gas conversion rate of 91.48 percent, equivalent to a specific activity of about 12.5. At the end of the 165.5 hour run these had deactivated steadily, with an apparent leveling off at the very end, to 68.49 percent and about 4.0, respectively.

Another noteworthy property was an extremely high water gas

shift activity. Initially 69 percent of the oxygen was being converted to CO₂. While this also decreased throughout the run to a final level of 26 percent, that was still twice as high as for any previous intimately contacted catalyst.

Production of methane was very high initially at 32 percent, decreased to a low of about 10 percent at 94.5 hours on stream, then rose again to 16 percent at the end of the run. The high initial value was probably due in part to the high H₂:CO ratio in the reactor resulting from the initial high water gas shift activity.

Production of C₅⁺ fluctuated rather irregularly.

The olefin content of the C₄'s, initially around 22 percent, rose quickly to about 60 percent at 47 hours on stream. Isomerization of the pentane was low throughout the run. The Schulz-Flory plots were non-linear for the first three samples, but except for the usual high methane, were linear for the remainder of the run.

This is an important catalyst for its demonstration of the potential for obtaining very high specific activity. The instability both in syngas conversion and in selectivity suggests that the nature of the catalyst may have changed drastically during the course of the run.

RUN 12200-06

111 H₂CO
300 PSIG
260°C

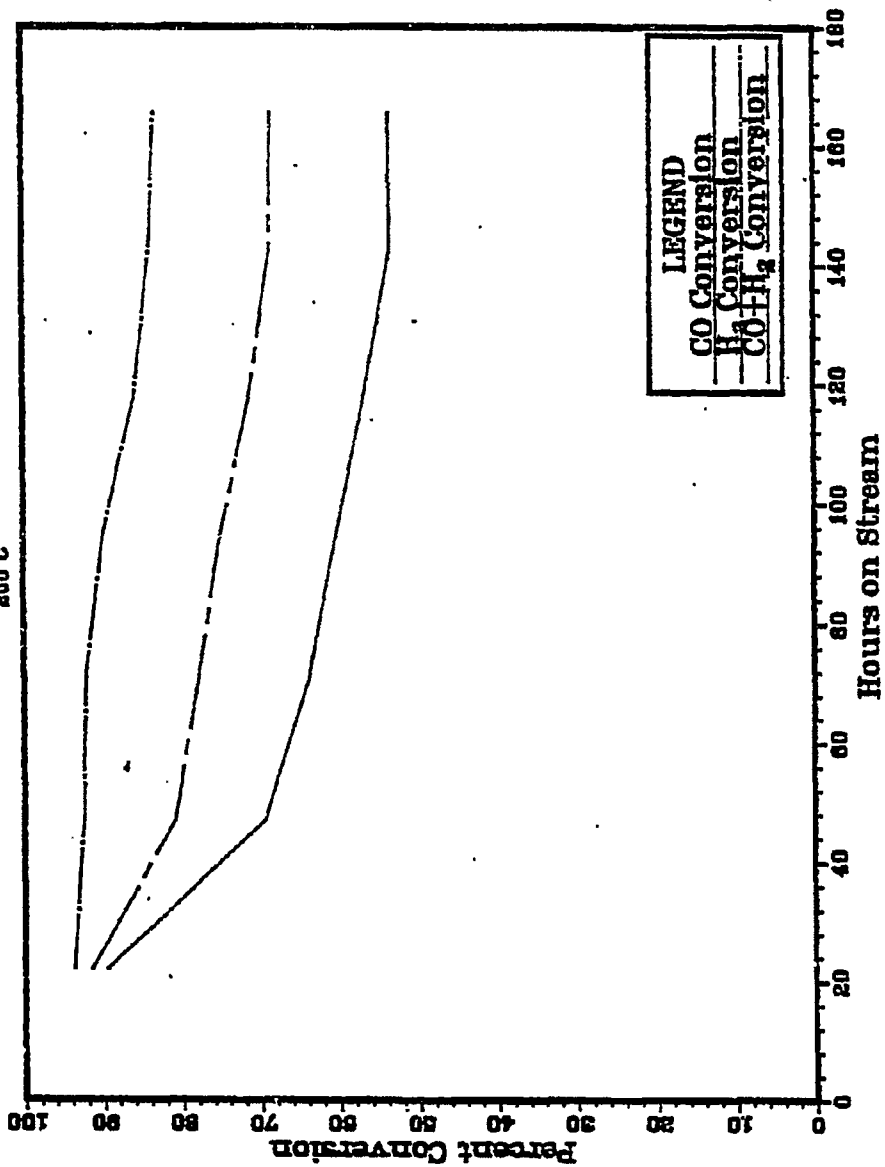


Fig. B29

RUN 12200-06

111 H₂CO
300 F/150
260°C

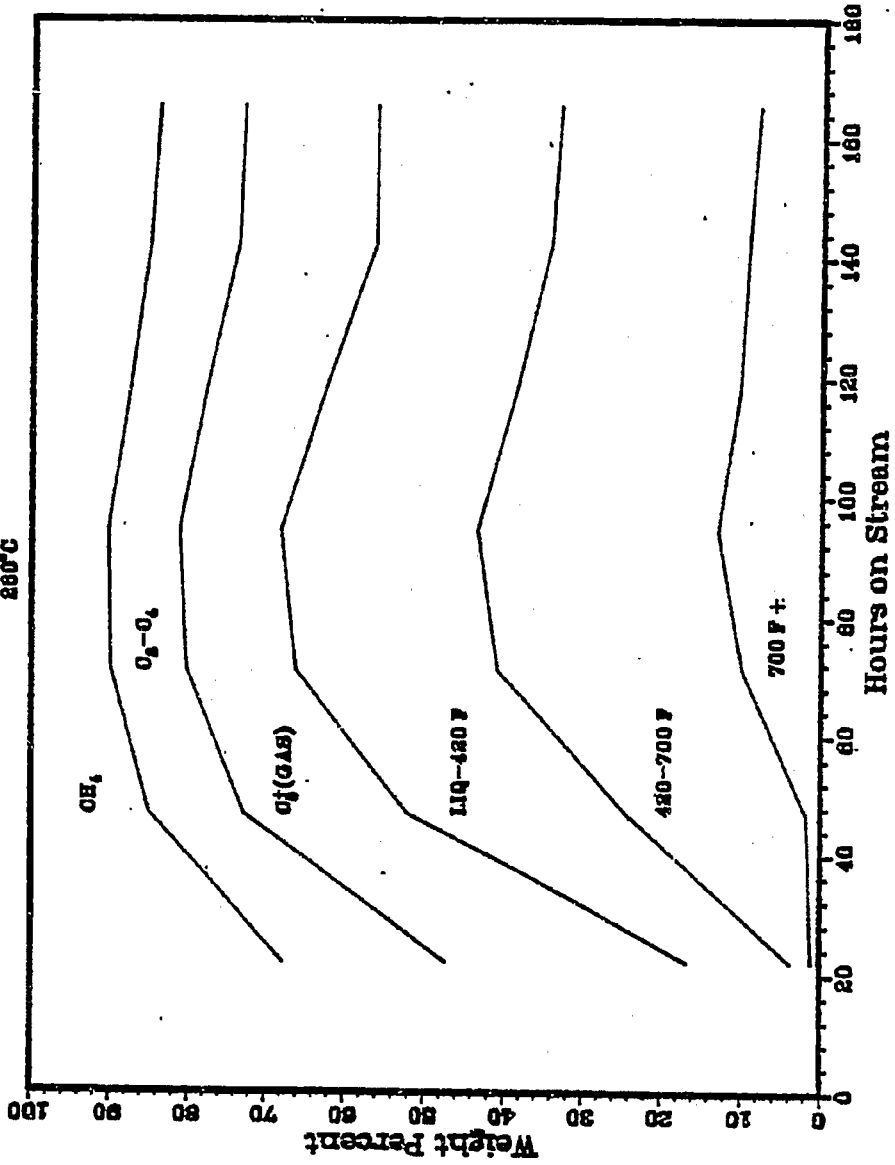


Fig. B30

RUN 12200-06

111 H₂O
300 PSIG
880°C

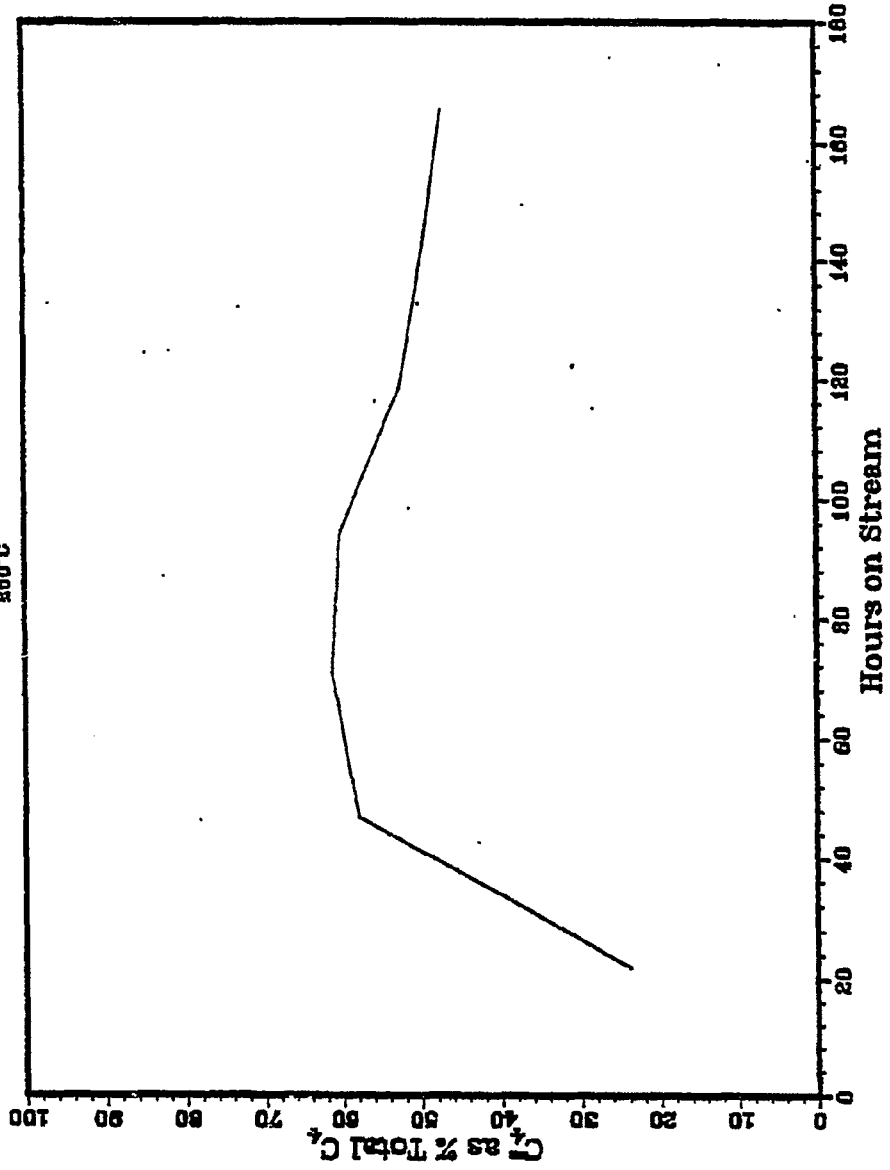


Fig. B31

RUN 12200-06

1:1 H₂:CO
900 PSIG
280°C

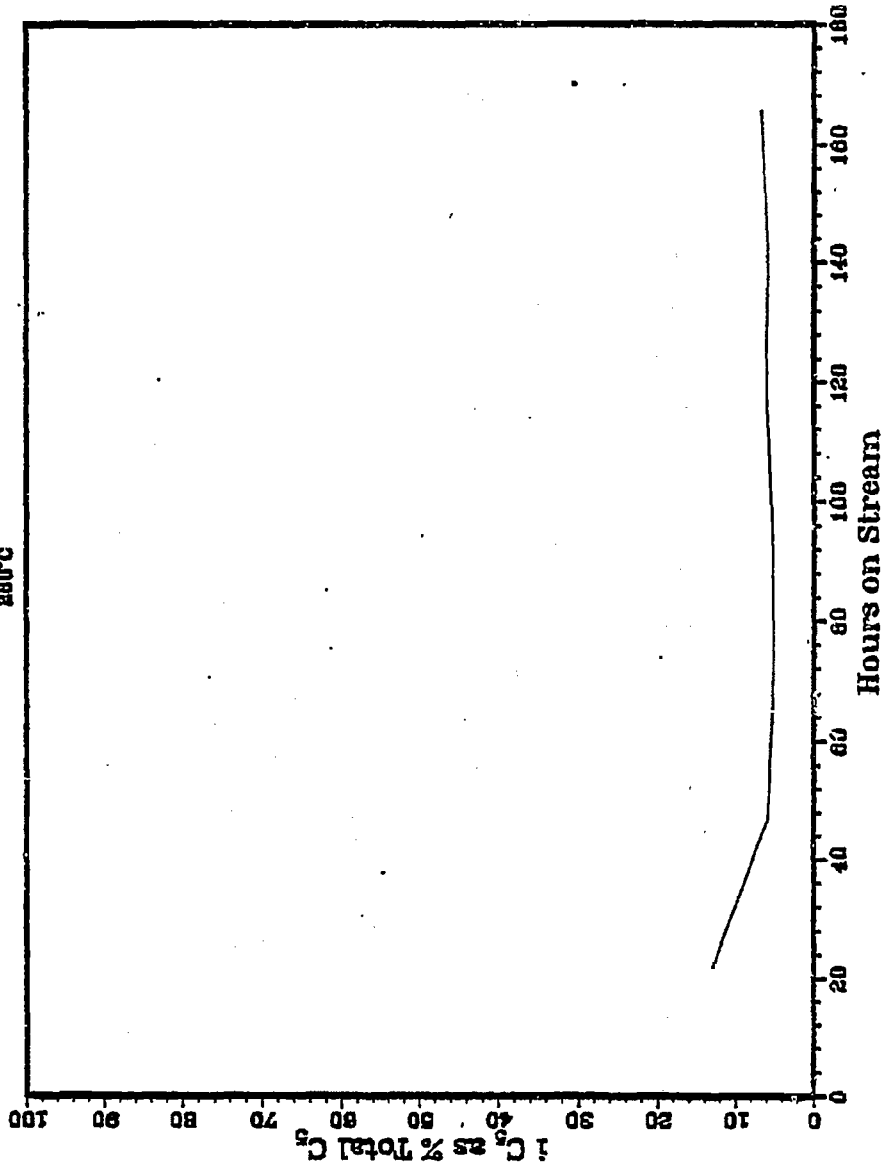


Fig. B32

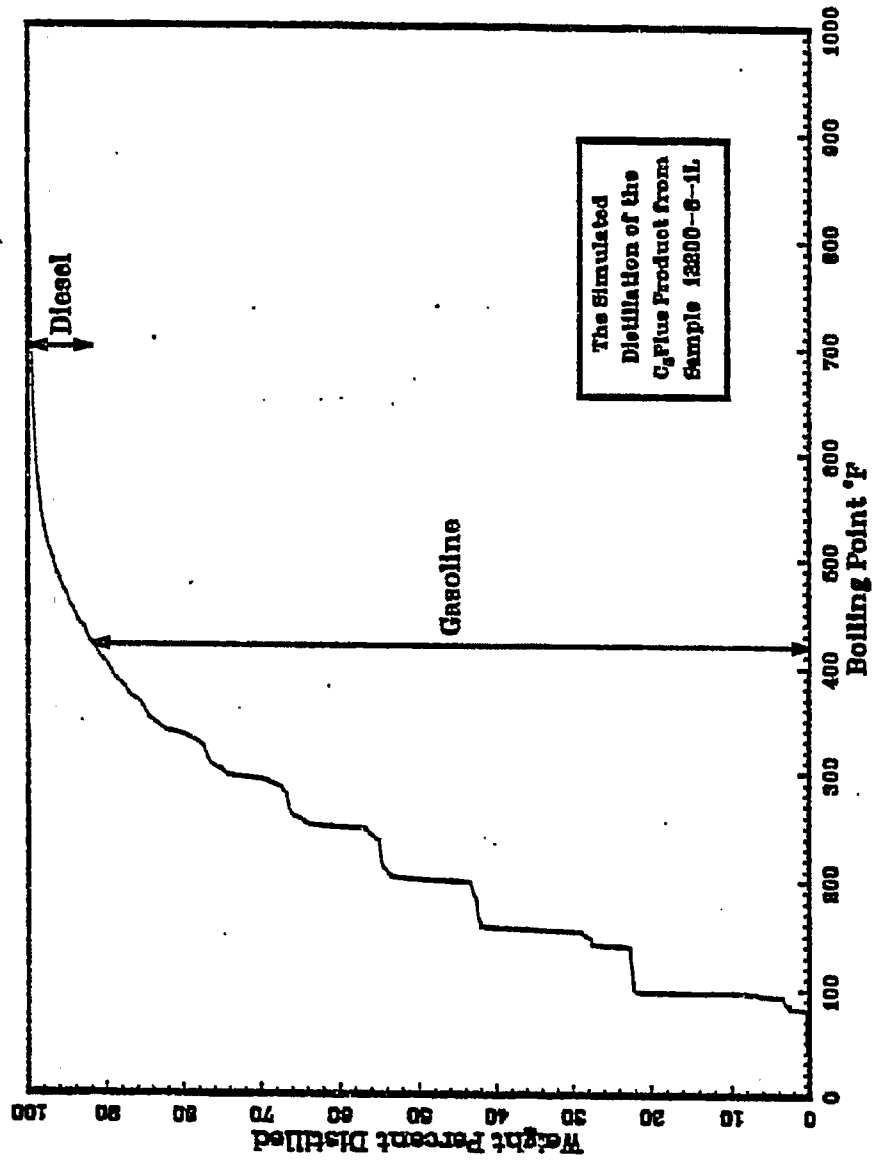


Fig. B33

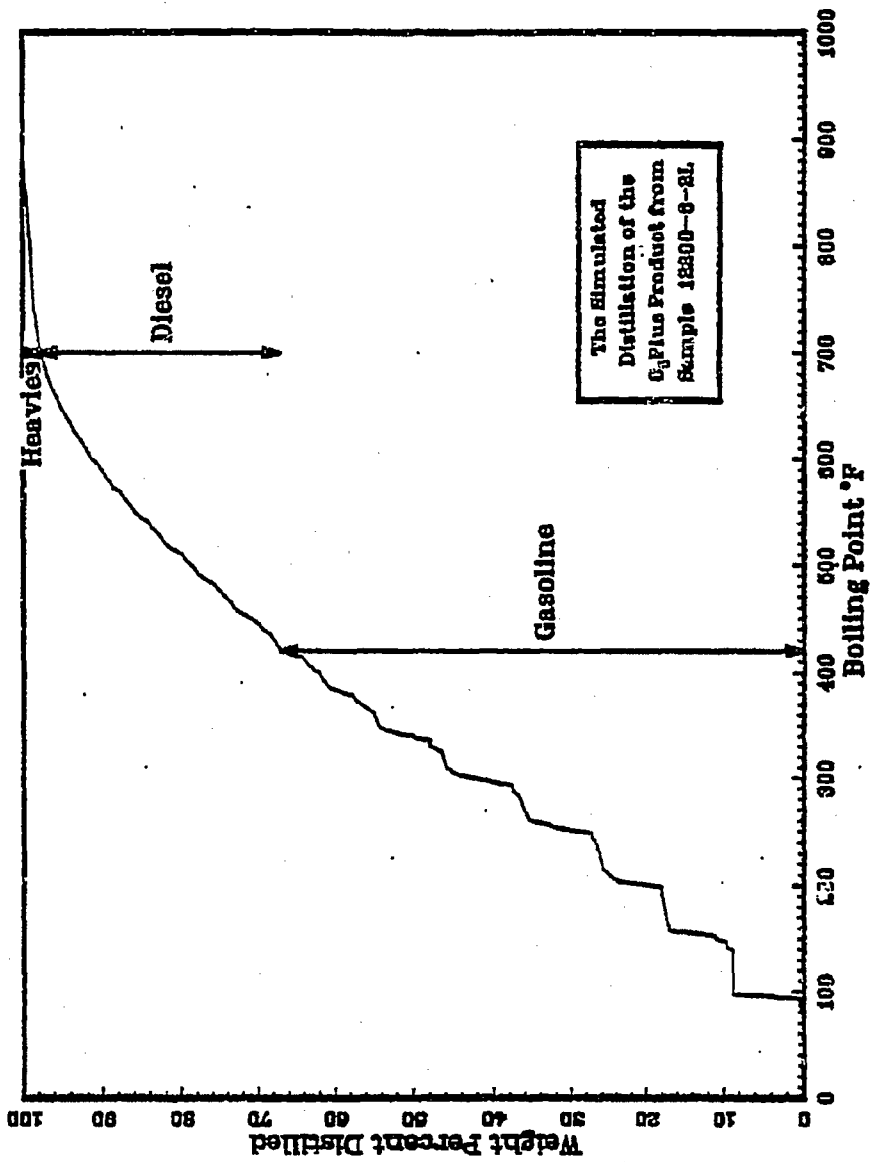


Fig. B34

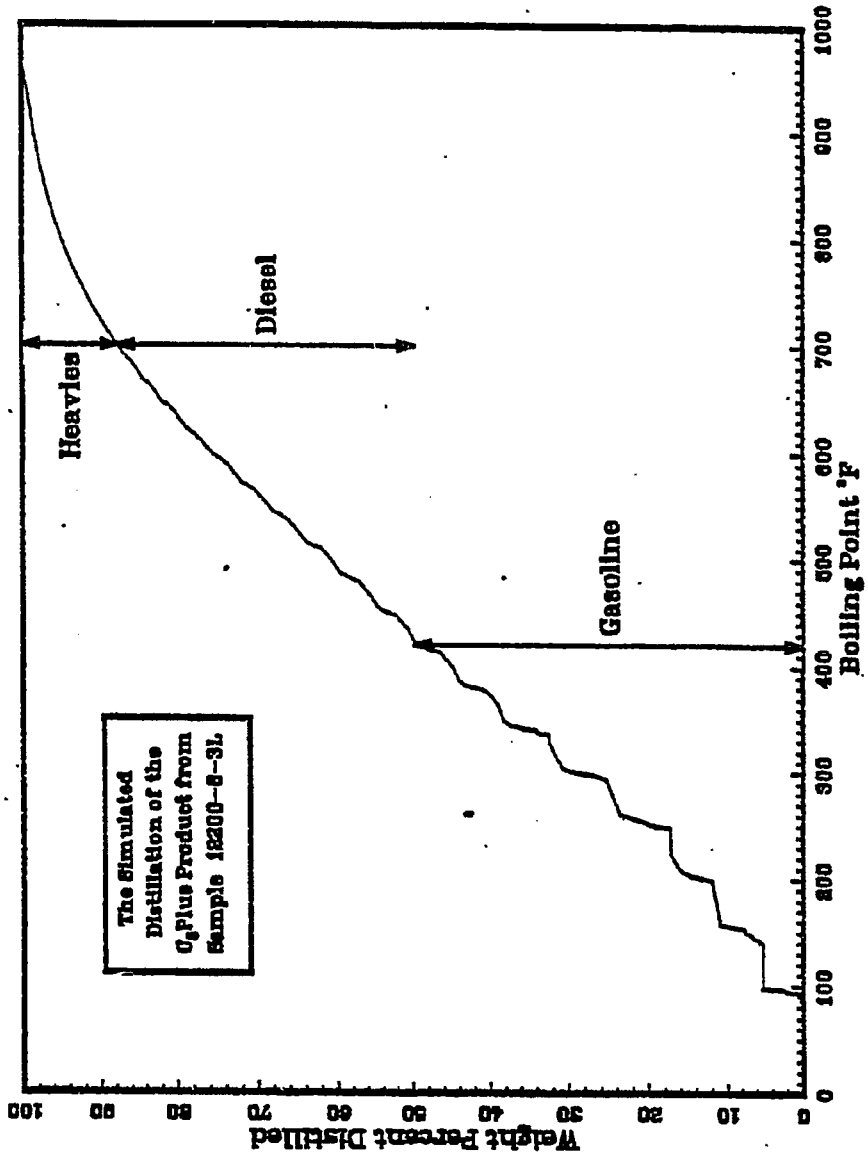
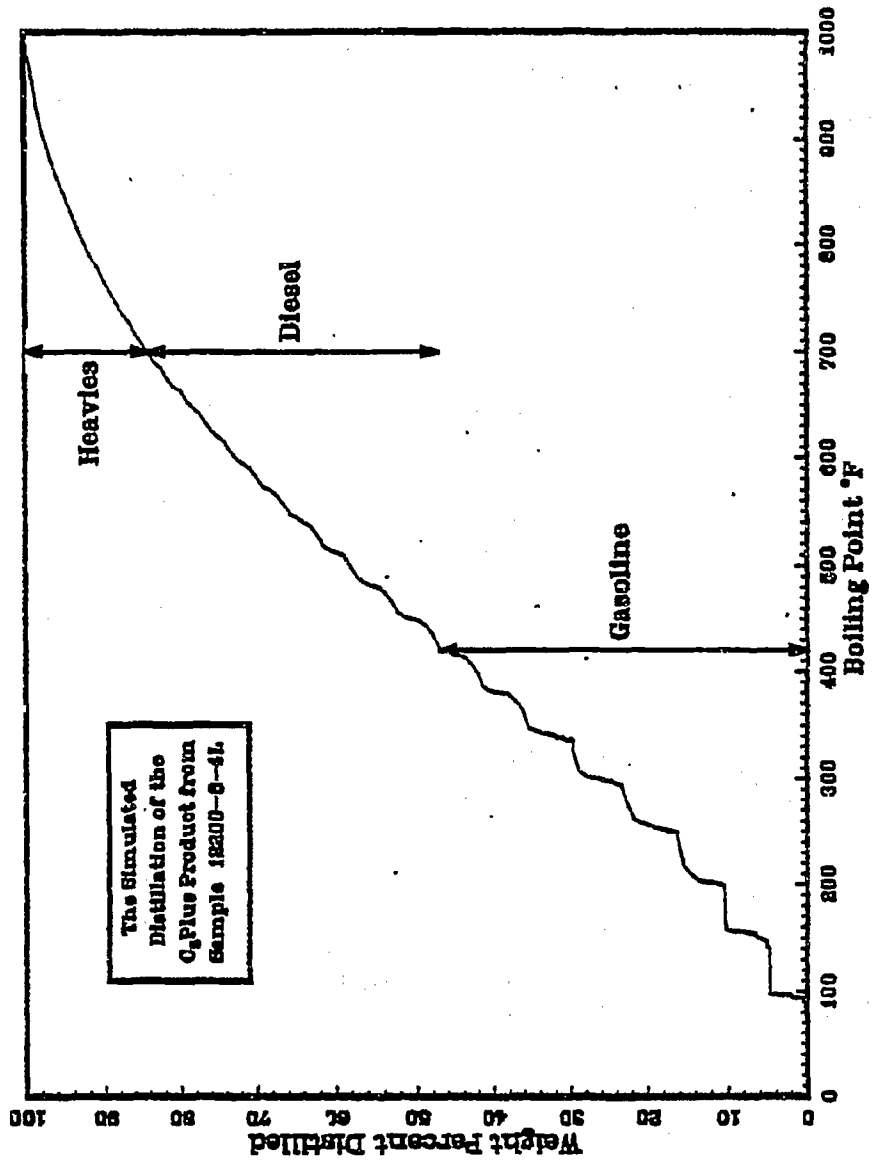
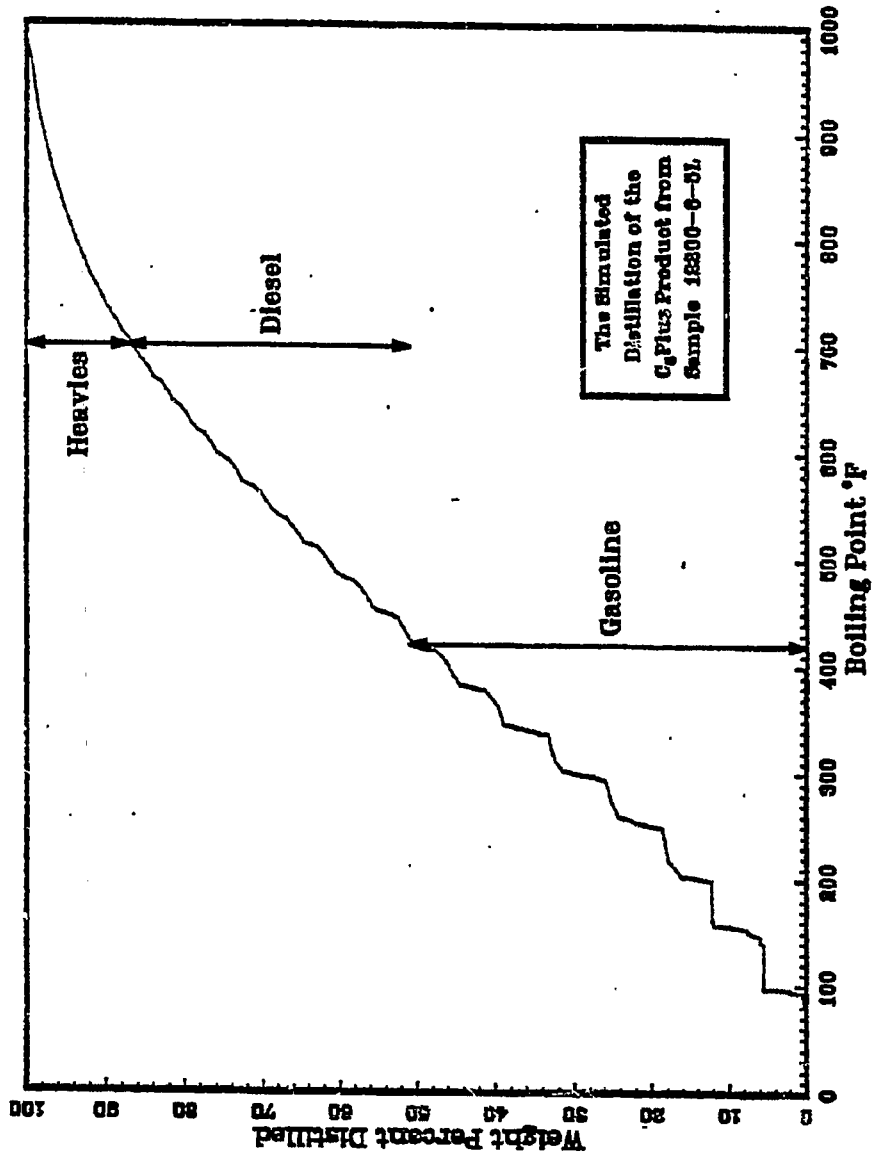


Fig. B35



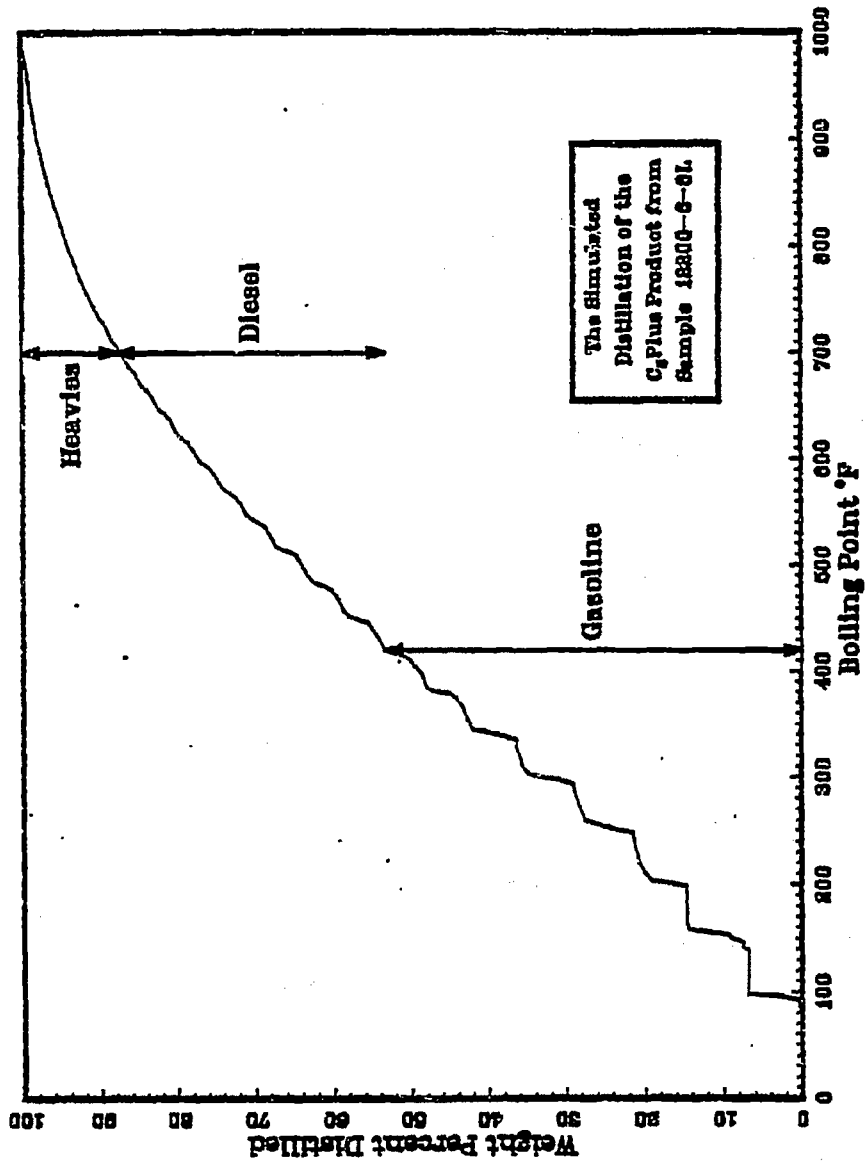
The Simulated
Distillation of the
C₁₅ Plus Product from
Sample 18200-8-41.

Fig. B36



The Simulated
Distillation of the
C₆ Plus Product from
Sample 18800-6-01.

Fig. B37



The Simulated
Distillation of the
C₁₂ Plus Product from
Sample 18200-6-81.

Fig. B38

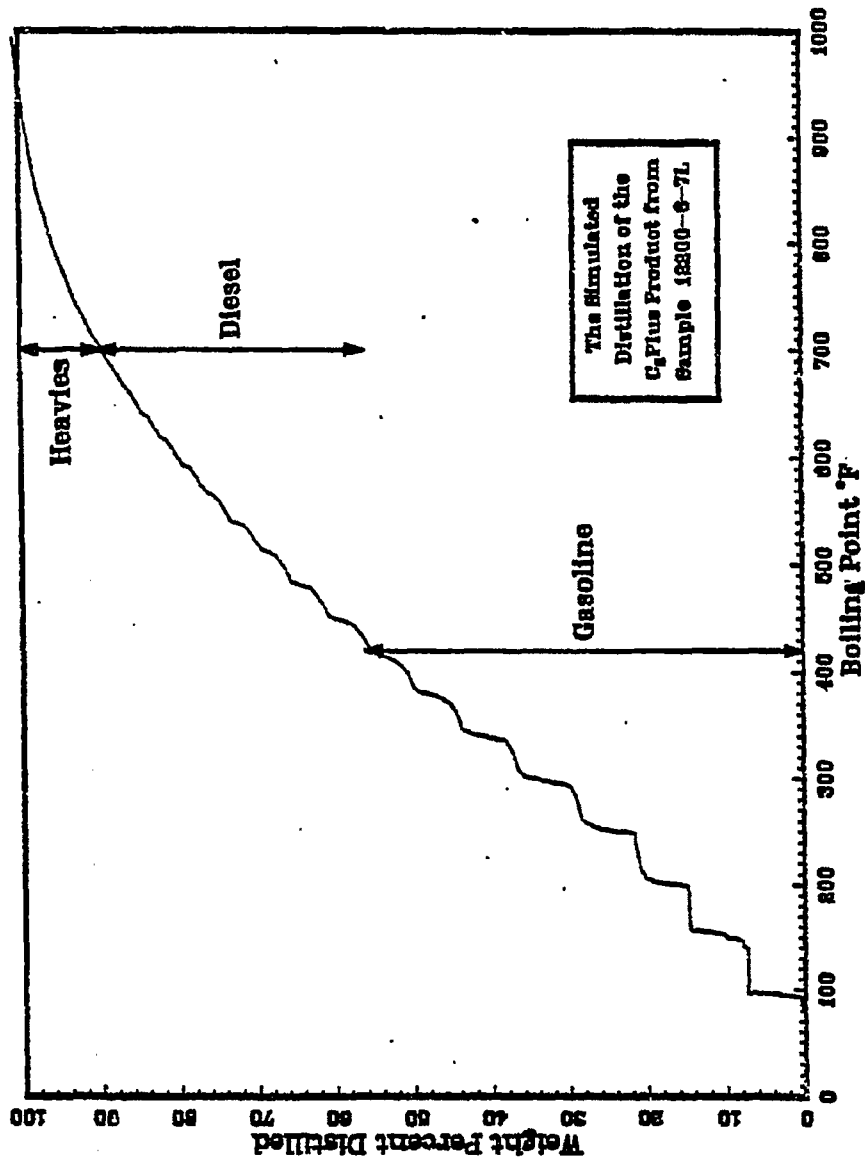


Fig. B39

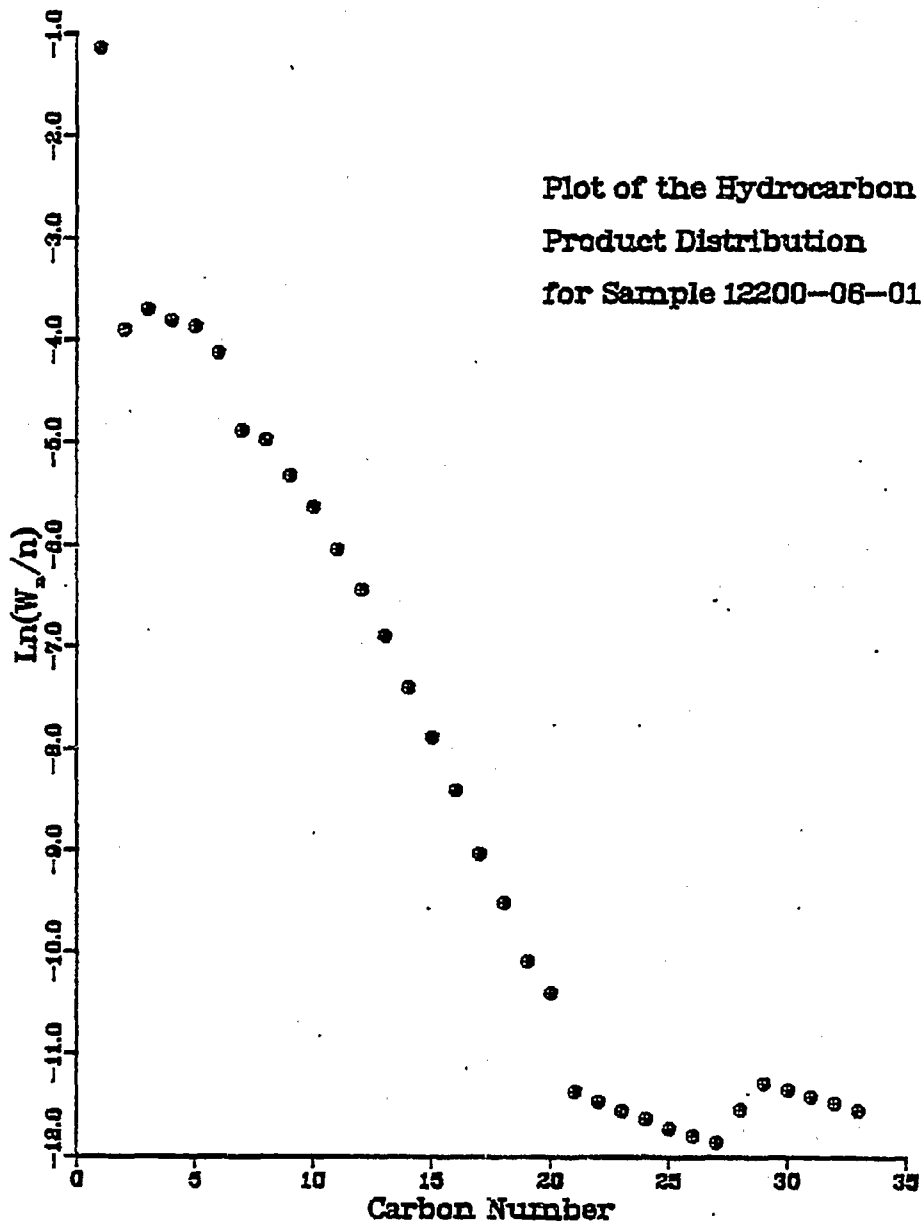


Fig. B40

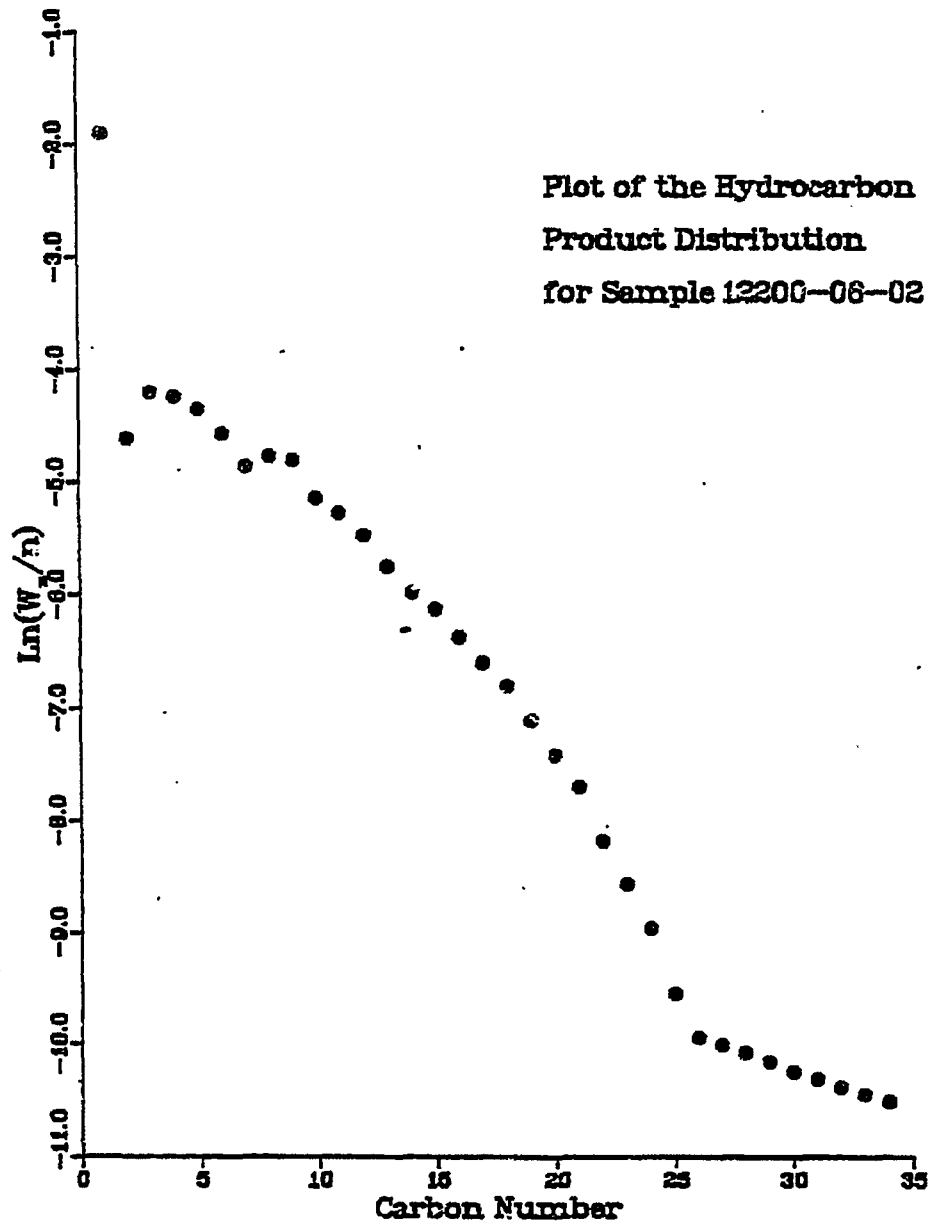


Fig. B41

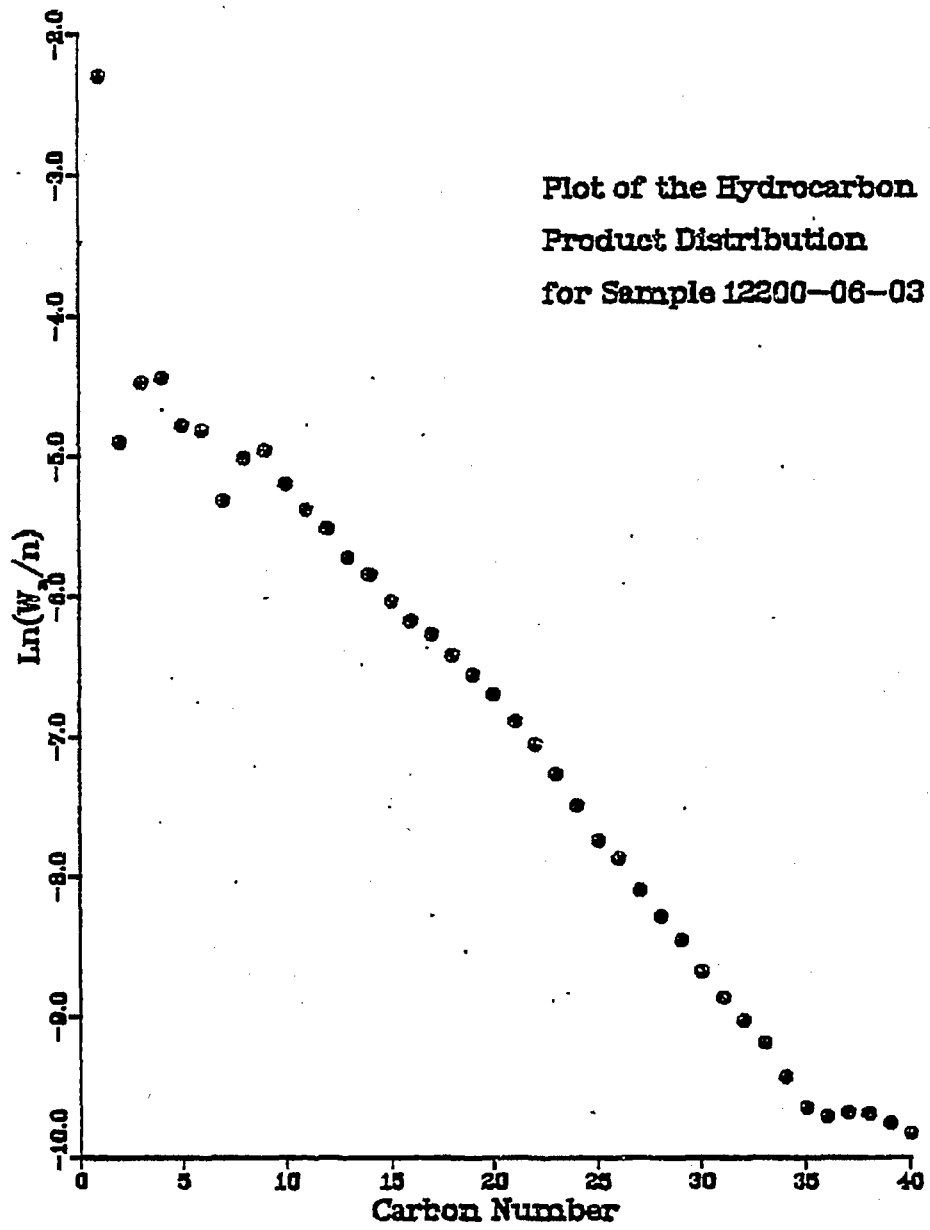


Fig. B42

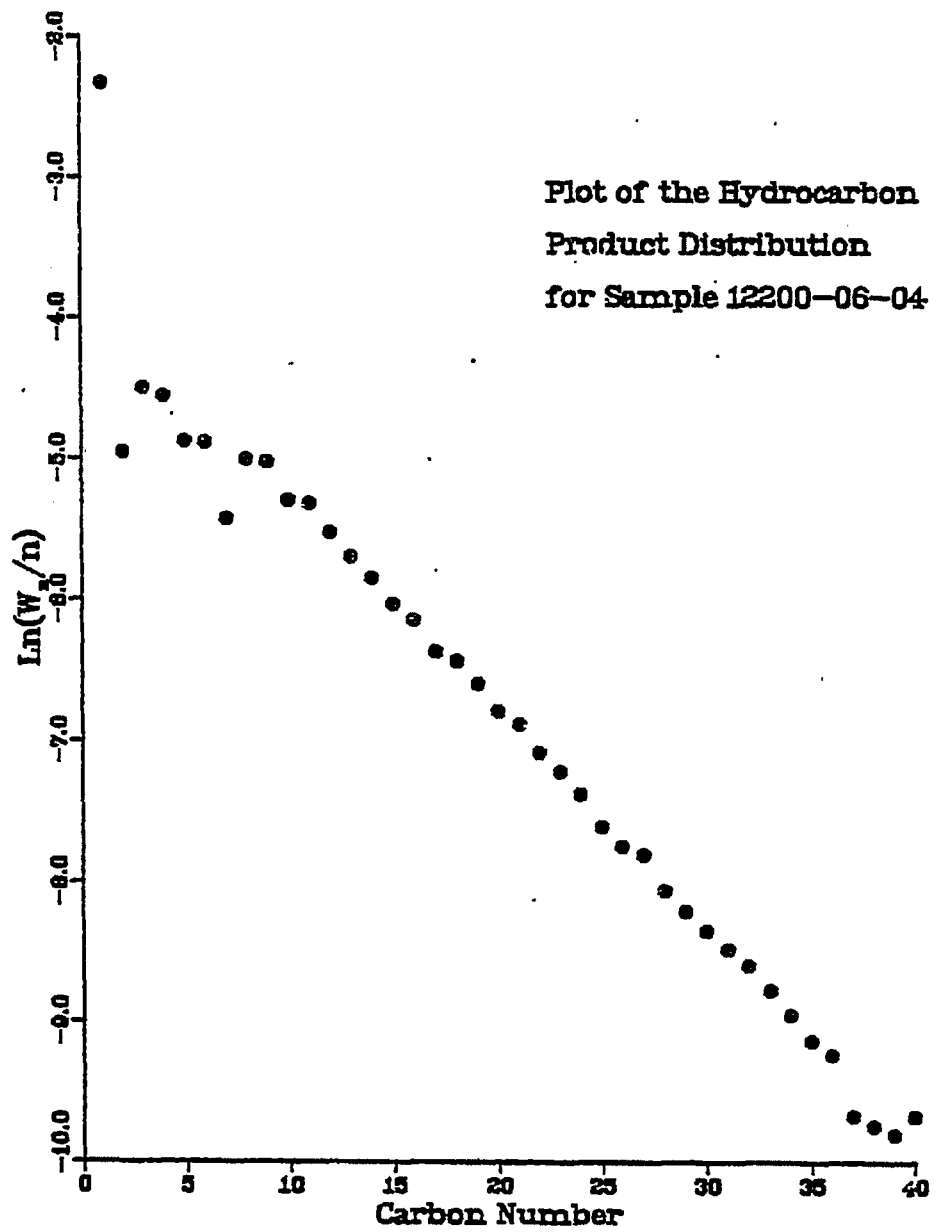


Fig. B43

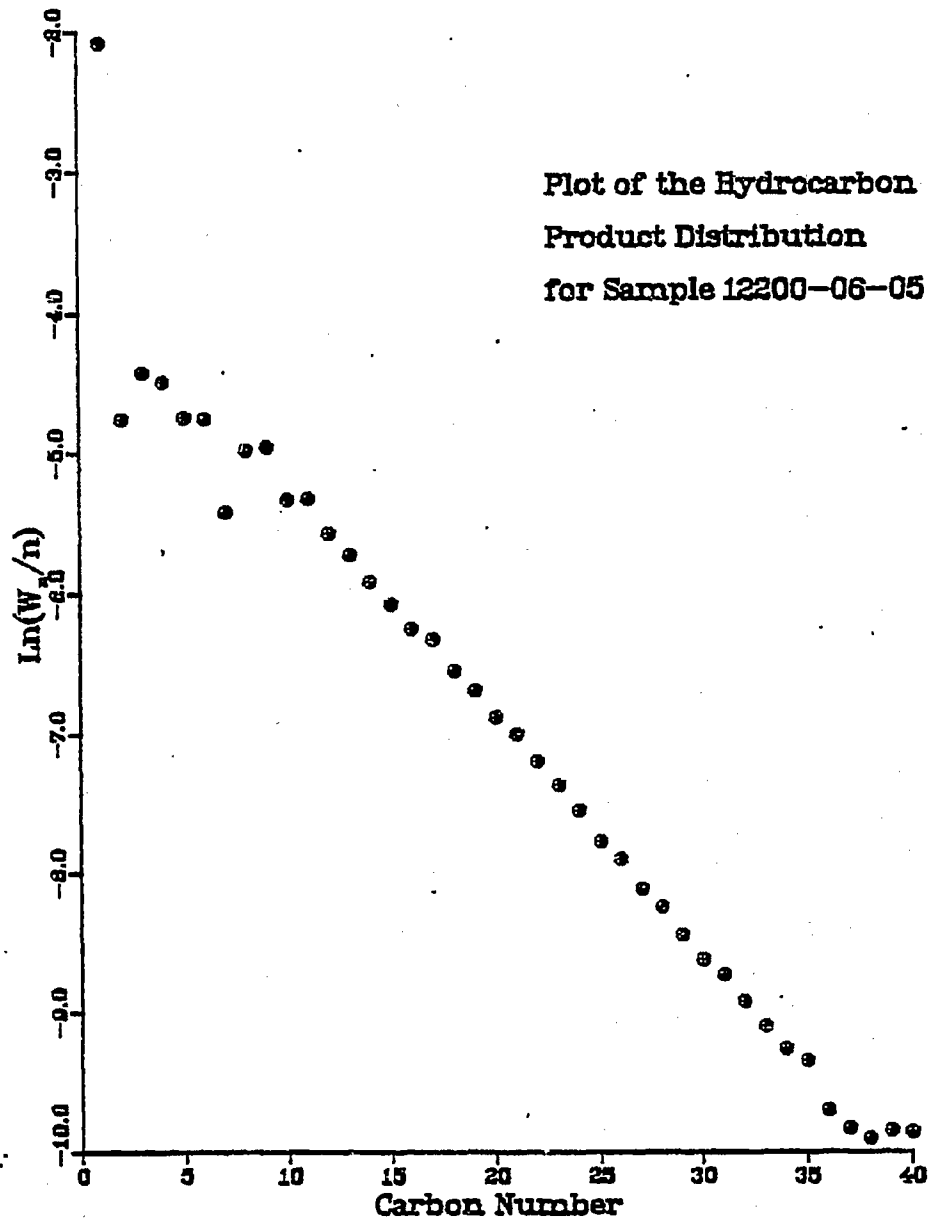


Fig. B44

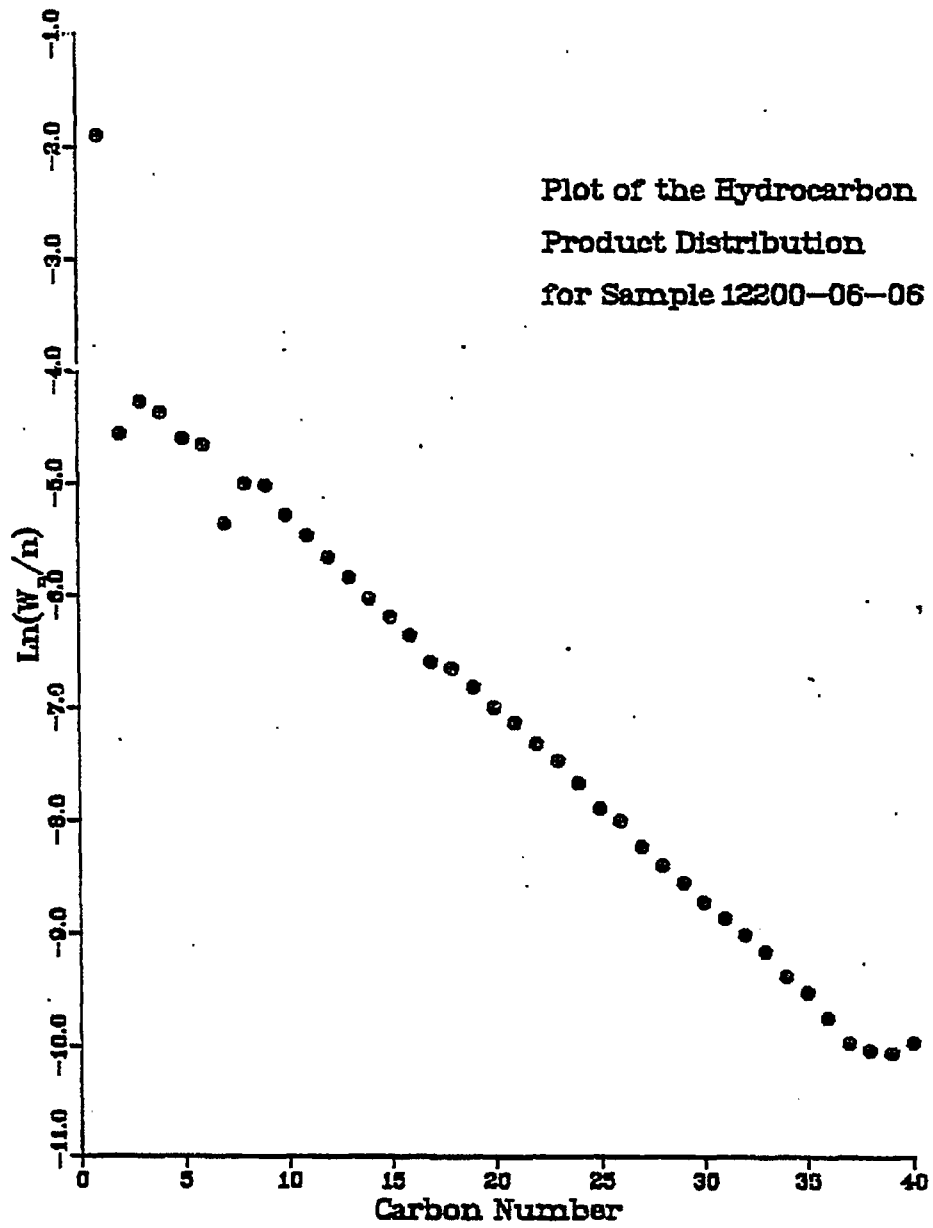


Fig. B45

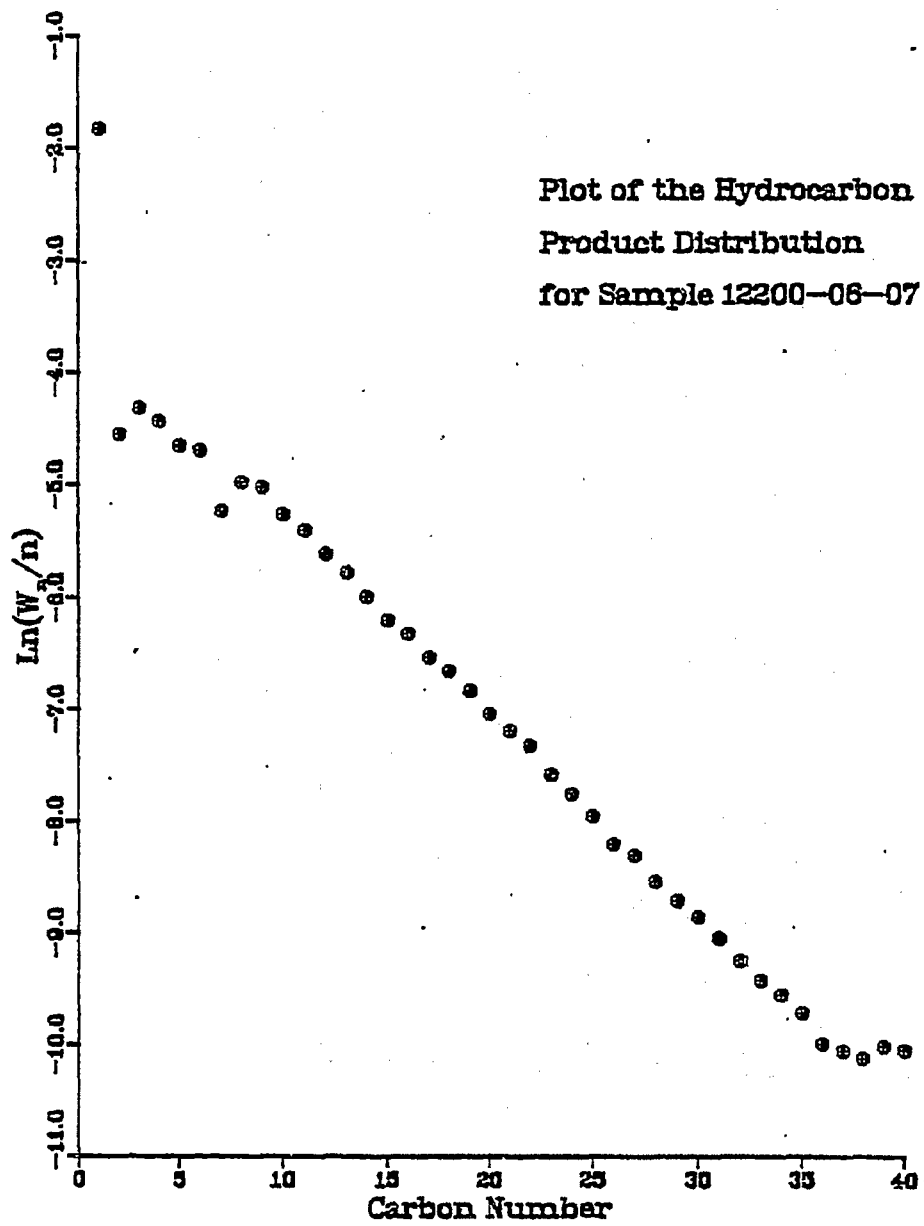
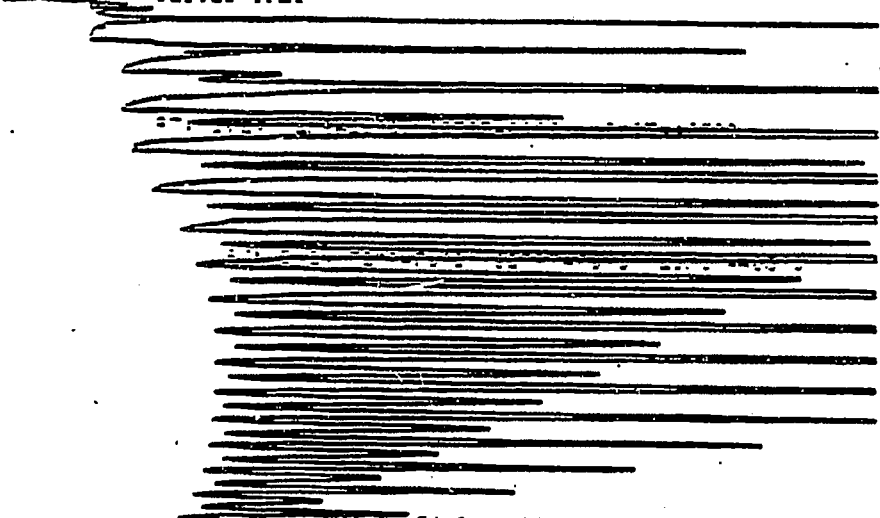


Fig. B46

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0000 0000 0000 0000 0000 0000

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12200-6-2L

Fig. B48

10T

ACT

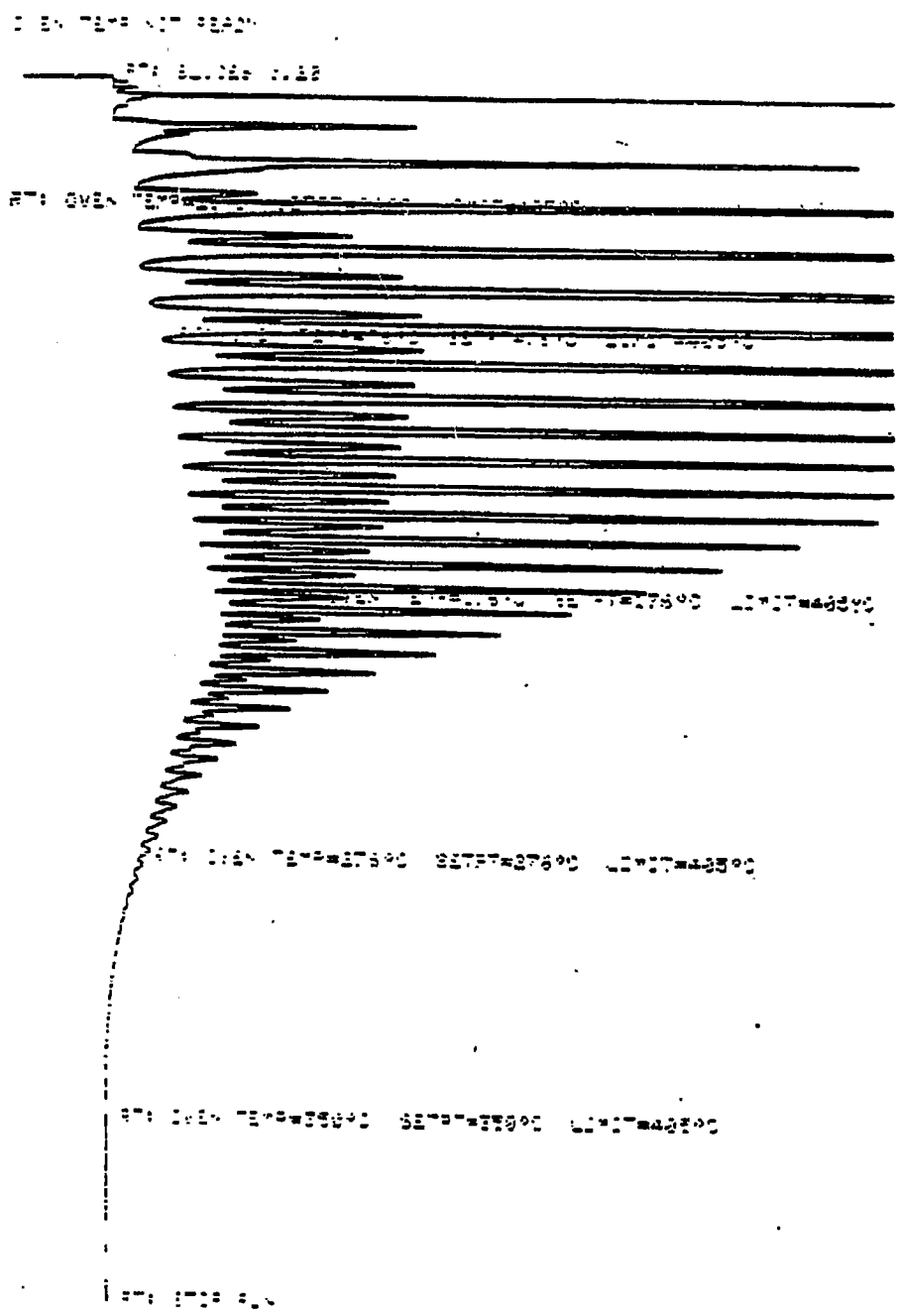


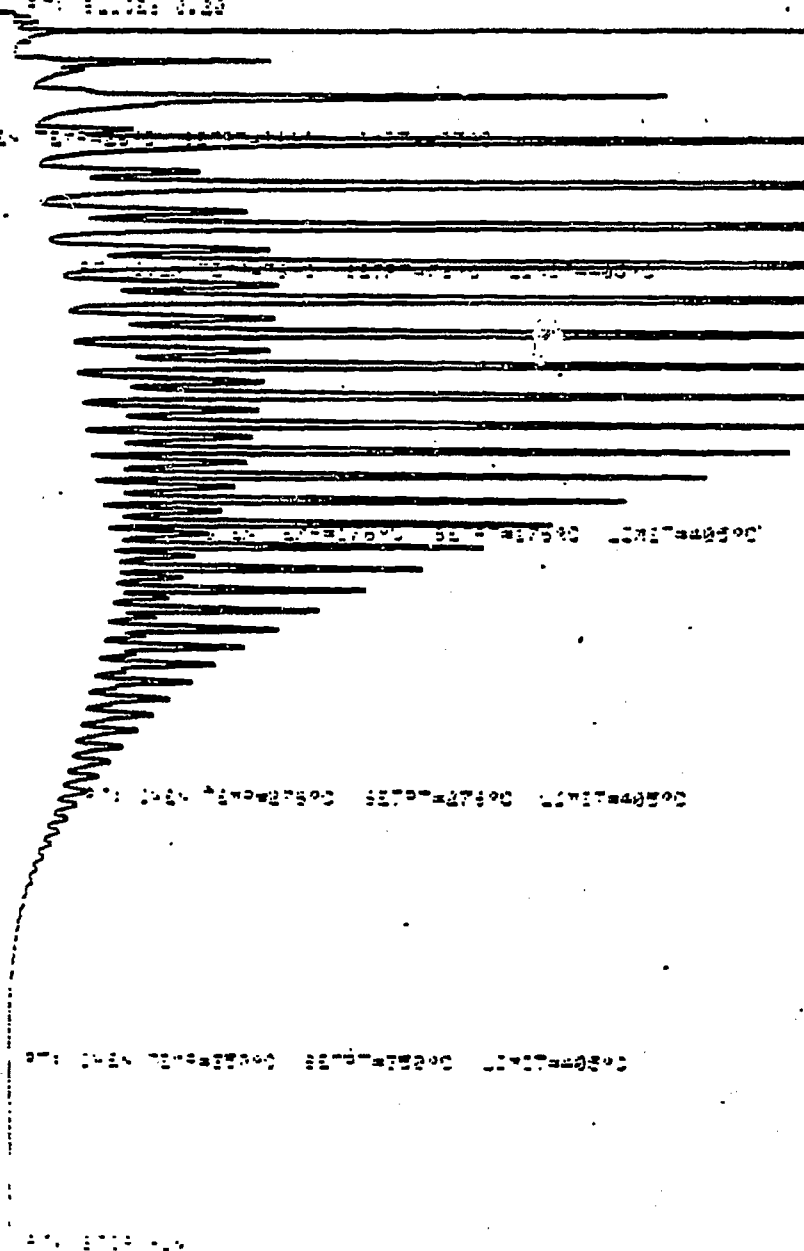
Fig. B49

GCT

12200-G-4L

12200-G-4L

12200-G-4L

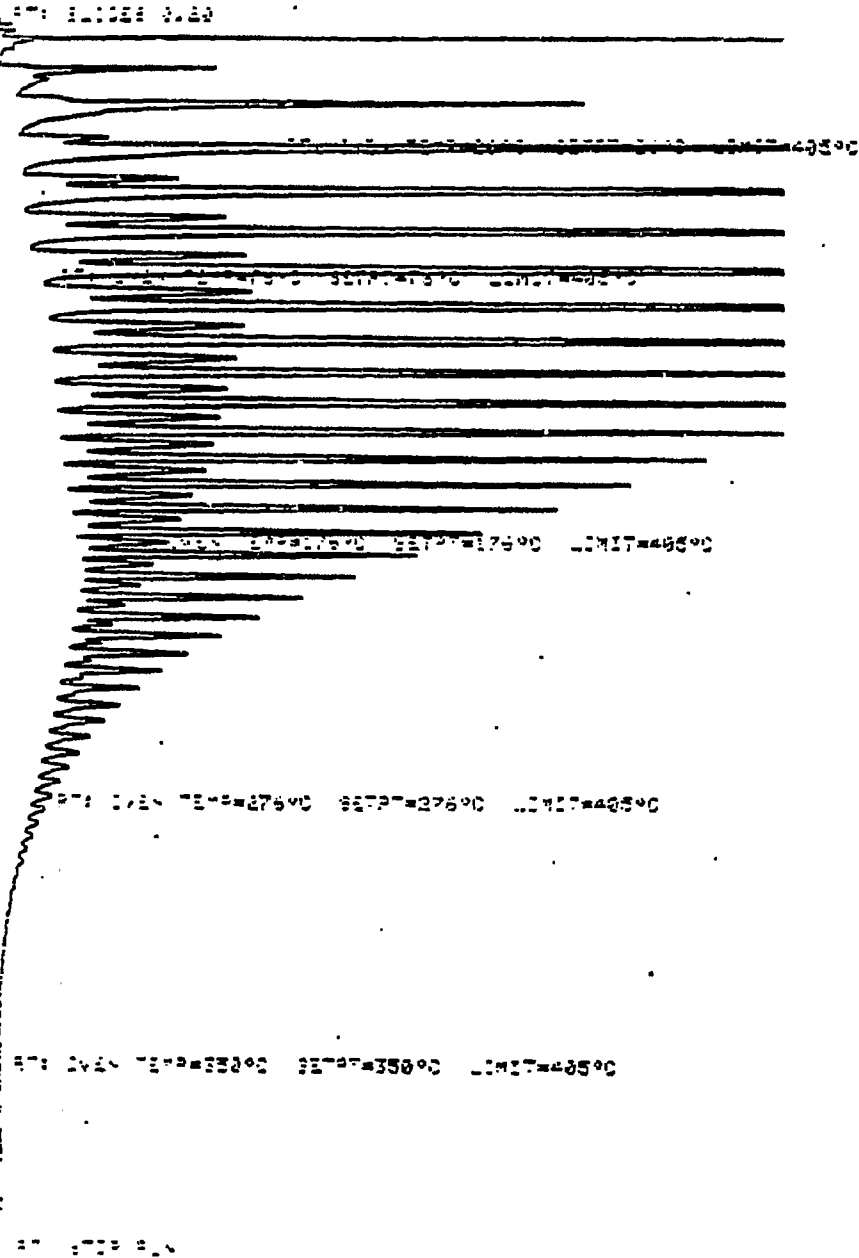


12200-G-4L

Fig. B50

OVER TIME OF TEST

TOT



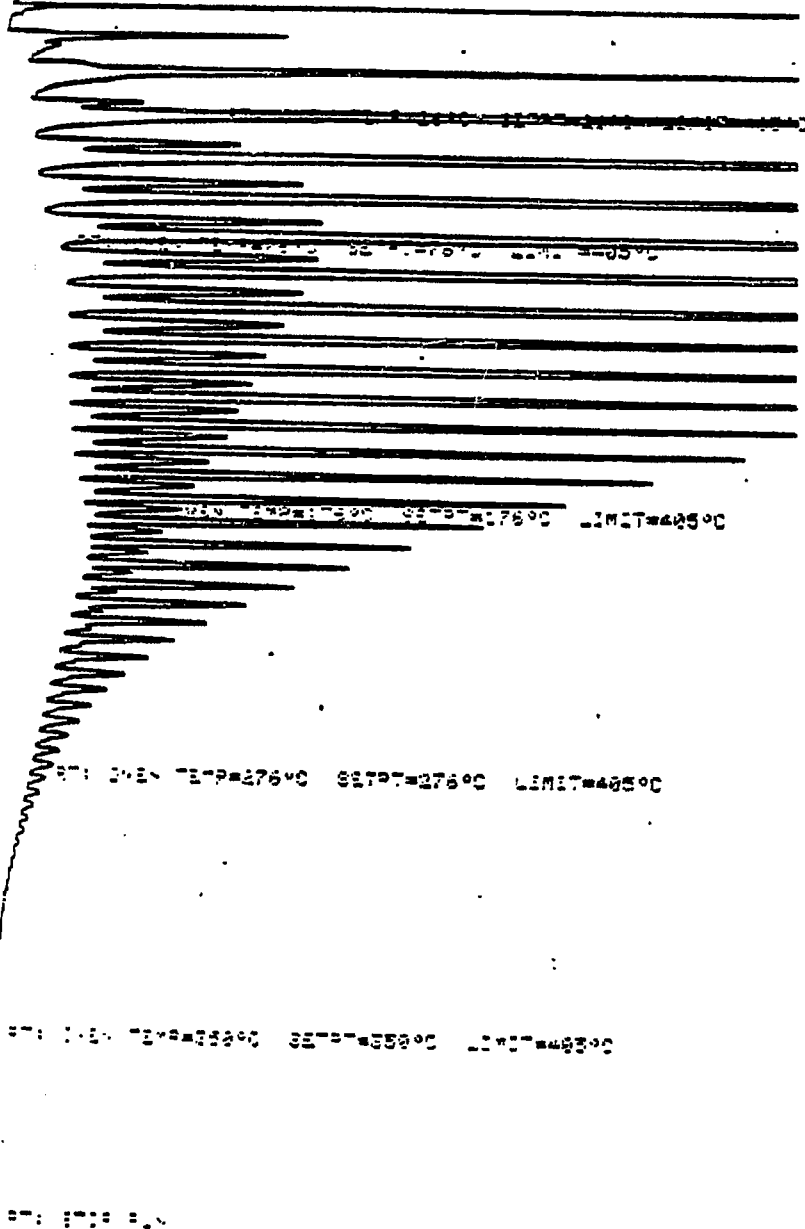
12200-6-51

Fig. B51

OVER TEMP NOT READY

ACT

ST: 12200-G-7L



ST: 12200-G-7L SETPT=275°C LIMIT=405°C

ST: 12200-G-7L SETPT=275°C LIMIT=405°C

ST: 12200-G-7L SETPT=350°C LIMIT=405°C

ST: 12200-G-7L

12200-G-7L
ST: 12200-G-7L

Fig. B53

RESULT OF SYNGAS OPERATION

RUN NO. 12200-06
 CATALYST CO-U103 12006-55 80 CC 35.06 GM (45.47 G AFTER RUN +9.41G)
 FEED H2:CO OF 50:50 @400 CC/MN OR 300 GHSV

RUN & SAMPLE NO.	12200-06-01	200-06-02	200-06-03	200-06-04	200-06-05
FEED H2:CO:AR	50:50: 0	50:50: 0	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	22.0	47.0	71.0	94.5	118.5
PRESSURE, PSIG	300	300	300	300	300
TEMP. C	261	261	261	261	260
FEED CC/MIN	400	400	400	400	400
HOURS FEEDING	22.00	25.00	24.00	23.50	24.00
EFFLNT GAS LITER	191.50	185.55	183.10	189.25	214.60
GM AQUEOUS LAYER	28.47	63.19	67.20	65.49	64.08
GM OIL	12.08	36.17	49.00	49.72	43.97
MATERIAL BALANCE					
GM ATOM CARBON %	91.47	84.85	92.02	93.92	93.69
GM ATOM HYDROGEN %	83.91	82.90	90.26	92.63	93.51
GM ATOM OXYGEN %	99.70	95.43	96.44	95.60	95.79
RATIO CHX/(H2O+CO2)	0.8447	0.7864	0.9078	0.9632	0.9515
RATIO X IN CHX	2.8007	2.4131	2.3169	2.3112	2.3660
USAGE H2/CO PRODT	0.9606	1.3003	1.4147	1.4679	1.5101
FEED H2/CO FRM EFFLNT	0.9173	0.9770	0.9808	0.9862	0.9980
RESIDUAL H2/CO RATIO	0.5495	0.2432	0.2159	0.2523	0.3282
RATIO CO2/(H2O+CO2)	0.6995	0.4027	0.3178	0.2833	0.2744
K SHIFT IN EFFLNT	1.2790	0.1639	0.1006	0.0997	0.1241
SPECIFIC ACTIVITY SA	12.5050	13.0794	12.3531	8.7423	5.5424
CONVERSION					
ON CO %	89.46	69.42	63.81	60.37	56.68
ON H2 %	93.68	92.39	92.03	89.86	85.75
ON CO+H2 %	91.48	80.77	77.78	75.01	71.20
PRDT SELECTIVITY, WT %					
CHA	32.13	15.02	10.09	9.78	12.47
C2 HC'S	4.05	1.97	1.49	1.41	1.71
C3H8	6.71	2.52	1.65	1.70	2.18
C3H6=	0.76	1.95	1.76	1.65	1.42
CAH10	6.85	2.46	1.86	1.71	2.15
CAH8=	2.08	3.26	2.84	2.50	2.31
CSH12	8.37	3.76	2.47	2.34	2.84
CSH10=	1.65	2.12	1.65	1.48	1.48
C6H14	8.81	4.45	3.03	2.70	3.24
C6H12= & CYCLO'S	0.58	1.21	1.07	1.16	1.31
C7+ IN GAS	11.26	9.19	5.82	5.39	6.36
LIQ HC'S	16.75	52.08	66.27	68.19	62.53
TOTAL	100.00	100.00	100.00	100.00	100.00

Table B3

SUB-GROUPING					
C1 -C4	52.58	27.19	19.70	18.75	22.24
C5 -420 F	43.52	48.44	39.35	37.74	39.05
420-700 F	2.80	22.60	31.01	30.55	28.39
700-END PT	1.11	1.77	9.94	12.96	10.32
C5+-END PT	47.42	72.81	80.30	81.25	77.76
ISO/NORMAL MOLE RATIO					
C4	0.0321	0.0113	0.0695	0.0122	0.0166
C5	0.1483	0.0617	0.0535	0.0550	0.0633
C6	0.3815	0.1431	0.1507	0.1039	0.1176
C4=	0.2132	0.0568	0.0584	0.0471	0.0607
PARAFFIN/OLEFIN RATIO					
C3	8.3698	1.2358	0.8958	0.9865	1.4682
C4	3.1793	0.7279	0.6313	0.6613	0.8999
C5	4.9340	1.7236	1.4525	1.5407	1.8674
SCHULZ-FLORY DISTRBTN					
ALPHA (EXP(SLOPE))	0.6845	0.7879	0.8653	0.8732	0.8616
RATIO CH4/(1-A)**2	3.2282	3.3395	5.5626	6.0800	6.5085
LIQ HC COLLECTION					
PHYS. APPEARANCE	CLD OIL	CLD OIL	CLD OIL	CLD OIL	CLD OIL
DENSITY	0.7287	0.7761	0.7942	0.7946	0.8108
N, REFRACTIVE INDEX	1.4110	1.4200	1.4274	1.4289	1.4274
SIMULT'D DISTILATN					
10 WT % @ DEG F	210	253	261	275	273
16	244	270	302	307	303
50	339	414	485	500	484
84	453	573	691	727	706
90	490	618	748	788	766
RANGE(16-84 %)	209	303	329	420	403
WT % @ 420 F	76.70	53.20	38.20	36.20	38.10
WT % @ 700 F	93.40	96.60	85.00	81.00	83.50

Table B3, cont

RESULT OF SYNGAS OPERATION

RUN NO. 12200-06
 CATALYST CO-U103 12006-55 80 CC 36.06 GM (45.47 G AFTER RUN +9.41 G)
 FEED H2:CO OF 50:50 @400 CC/MM OR 300 GHSV

RUN & SAMPLE NO. 12200-06-06 200-06-07

	=====	=====
FEED H2:CO:AR	50:50: 0	50:50: 0
HRS ON STREAM	142.5	165.5
PRESSURE, PSIG	300	300
TEMP. C	261	261

FEED CC/HIN	400	400
HOURS FEEDING	24.00	23.00
EFFLNT GAS LITER	230.60	223.90
GM AQUEOUS LAYER	64.22	61.18
GM OIL	37.24	36.41

MATERIAL BALANCE

GM ATOM CARBON %	93.95	94.30
GM ATOM HYDROGEN %	93.35	95.43
GM ATOM OXYGEN %	98.65	97.70
RATIO CHX/(H2O+CO2)	0.8913	0.9204
RATIO X IN CHX	2.4173	2.4376
USAGE H2/CO PRODT	1.5555	1.5688
FEED H2/CO FRM EFFLNT	0.9936	1.0121
RESIDUAL H2/CO RATIO	0.3459	0.3677
RATIO CO2/(H2O+CO2)	0.2703	0.2639
K SHIFT IN EFFLNT	0.1281	0.1318
SPECIFIC ACTIVITY SA	4.3789	4.0024

CONVERSION

ON CO %	53.55	53.64
ON H2 %	83.83	83.16
ON CO+H2 %	68.64	68.49

PRDT SELECTIVITY, WT %

CHA	14.97	16.07
C2 HC'S	2.09	2.11
C3H8	2.69	2.69
C3H6=	1.50	1.32
CAH10	2.59	2.56
C4H8=	2.45	2.20
C5H12	3.43	3.32
C5H10=	1.61	1.45
C6H14	3.82	3.66
C6H12= & CYCLO'S	1.37	1.21
C7+ IN GAS	7.15	7.04
LIQ HC'S	56.35	56.36

TOTAL	100.00	100.00
-------	--------	--------

Table B4

SUB-GROUPING		
C1 -C4	26.28	26.95
C5 -420 F	39.35	39.85
420-700 F	25.02	25.14
700-END PT	9.35	8.06
C5+-END PT	73.72	73.05
ISO/NORMAL MOLE RATIO		
C4	0.0171	0.0229
C5	0.0618	0.0721
C6	0.1249	0.1256
C4=	0.0691	0.0830
PARAFFIN/OLEFIN RATIO		
C3	1.7122	1.9454
C4	1.0178	1.1198
C5	2.0749	2.2226
SCHULZ-FLORY DISTRBTN		
ALPHA (EXP(SLOPE))	0.8523	0.8479
RATIO CH4/(1-A)**2	6.8602	6.9509
LIQ HC COLLECTION		
PHYS. APPEARANCE	CLD OIL	CLD OIL
DENSITY (* 40 C)	0.7480*	0.7471*
N, REFRACTIVE INDEX	1.4276*	1.4210*
SIMULT'D DISTILATN		
10 WT % @ DEG F	273	262
16	303	301
50	482	472
84	706	685
90	767	746
RANGE(16-84 %)	403	384
WT % @ 420 F	39.00	41.10
WT % @ 700 F	83.40	85.70

Table B4, cont

IV. Run 12 (12185-07) with Catalyst 12 (Co/Th/X₄/UCC-103+UCC-101)

This run was an attempt to reproduce the results of Run 11677-11 of the Third Annual Report of the previous contract. The catalyst was formulated in the same way as for that run. The thorium-promoted cobalt oxide was formed in close contact with UCC-103, then further promoted with X₄. The resulting powder was mixed with UCC-101 in a weight ratio of 1.125:1, and the mixture, after bonding with 15 weight percent silica, was extruded as 1/8-inch pellets. The final catalyst contained 4.4 percent cobalt, 0.6 percent thorium and 0.4 percent X₄.

Conversion, product selectivity, isomerization of the pentane, and percent olefins of the C₄'s are plotted against time on stream in Figs. B54-57. Simulated distillations of the C₅⁺ product are plotted in Figs. B58-70. Carbon number product distributions are plotted in Figs. B71-83. Chromatograms from simulated distillations are reproduced in Figs. B84-96. Detailed material balances appear in Tables B5-7.

The initial activity was comparable to that of Catalyst 11677-11. The initial syngas conversion was 50.2 percent for a specific activity of 1.2; the values for Catalyst 11677-11 were 55.5 percent and 0.9 respectively.

The water gas shift activity, however, was only one-third that of Catalyst 11677-11, with 3 percent of the oxygen converted

to CO₂ as against 9 percent.

In this run the stability was also inferior. With Catalyst 11677-11 there was essentially no deactivation throughout the 284.5 hours of the run, whereas this catalyst deactivated at a rate of one percentage point every 74 hours on stream. The difference may be due to an observed difference, determined by elemental analysis, in the ratios of cobalt to thorium:

Run 11677-11, ratio cobalt:thorium	27.0:1
Run 12185-07, " " "	47.6:1

The initial product selectivity, like the initial activity, was similar to that of Catalyst 11677-11, both runs producing about 13-14 percent methane and about 68 percent C₅⁺. The selectivity was similarly stable as well, with methane production increasing at one percentage point every 1300 hours and C₅⁺ production decreasing at one percentage point every 572 hours. In comparison with a mathematical model calculated for Catalyst 11677-11, however, the methane production of this catalyst was significantly lower, with a ratio of experimental to calculated methane of about 0.7:1 as against about 1:1 for Catalyst 11677-11.

Except for the loss of stability in this run, possibly due to the different cobalt:thorium ratios, and the lower water gas shift activity, the two runs compare reasonably well.

RUN 12185-07

111 H₂O
300 PSIG
260°C

600 PSIG

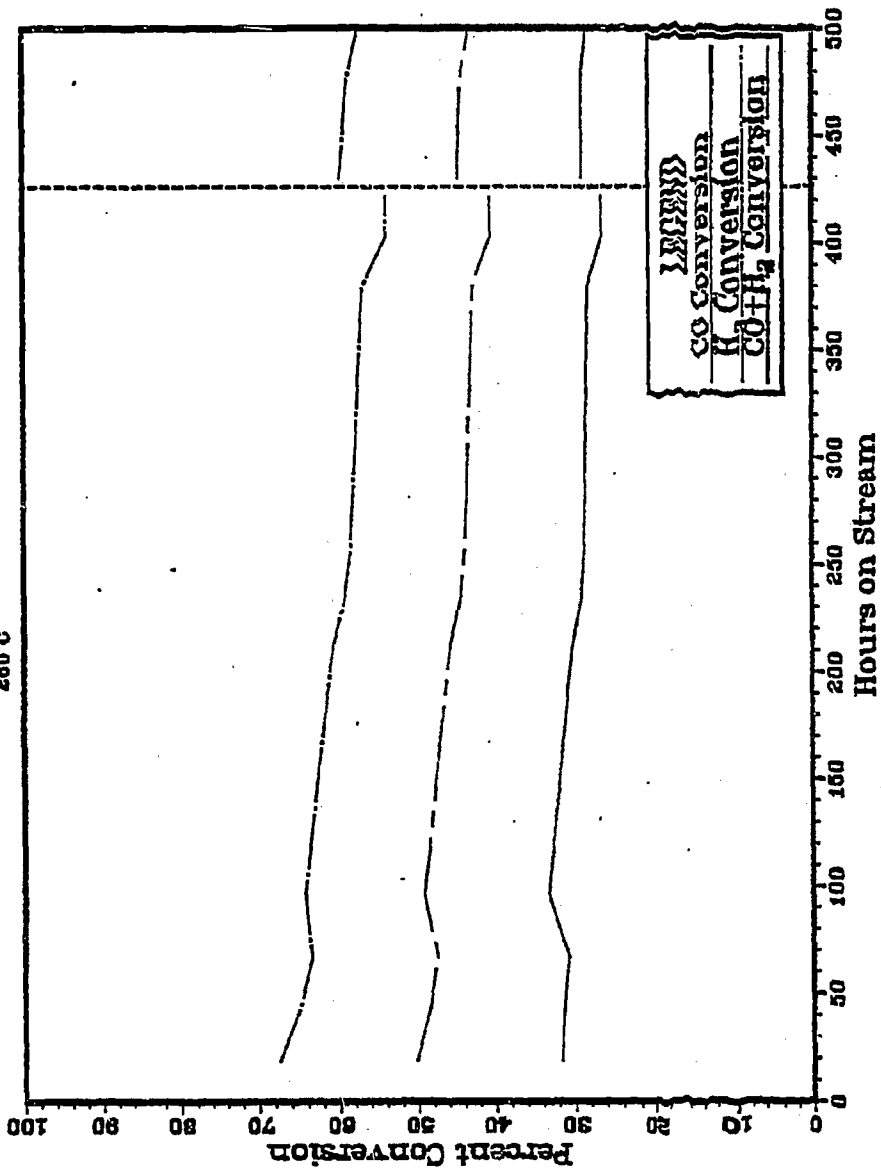


Fig. B54

RUN 12185-07

1:1 H₂:CO
300 PSIG
260°C

500 PSIG

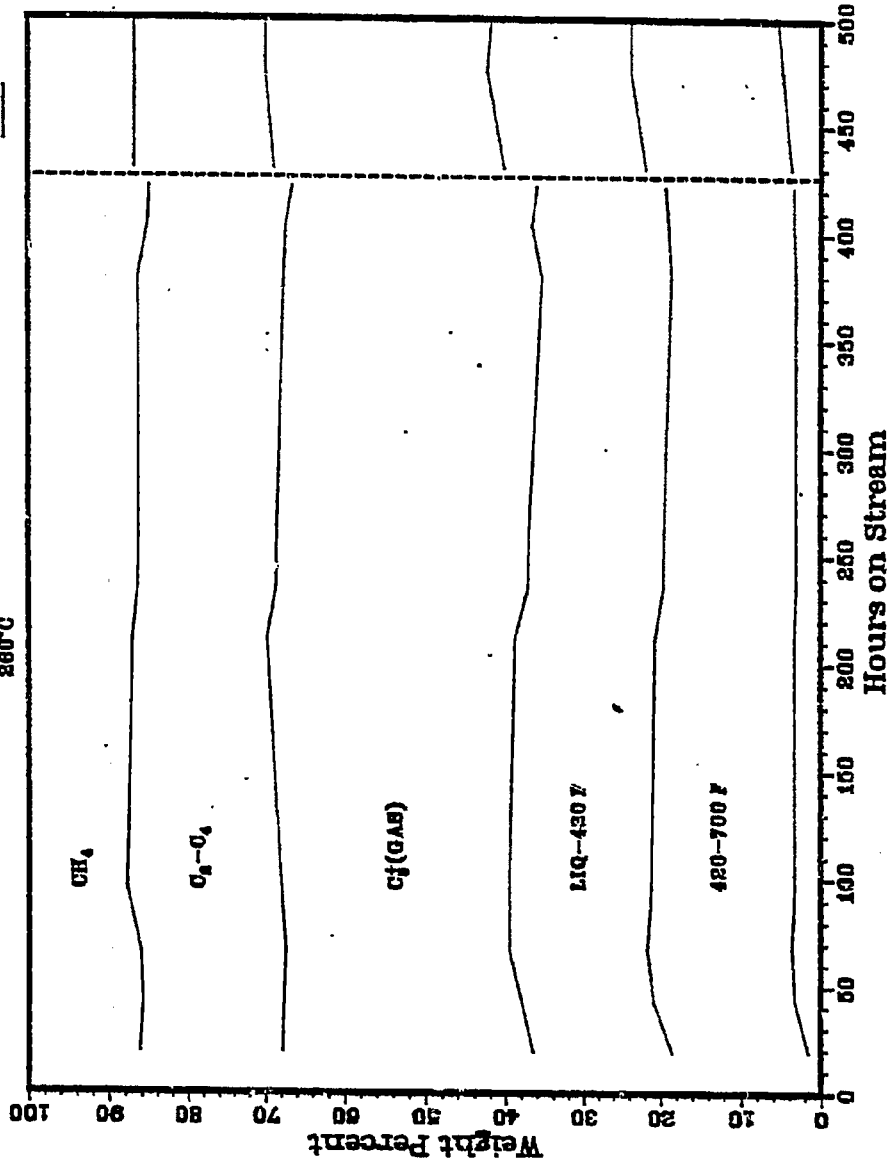


Fig. B55

RUN 12185-07

1117.100
300 PSIG
860°C

500 PSIG

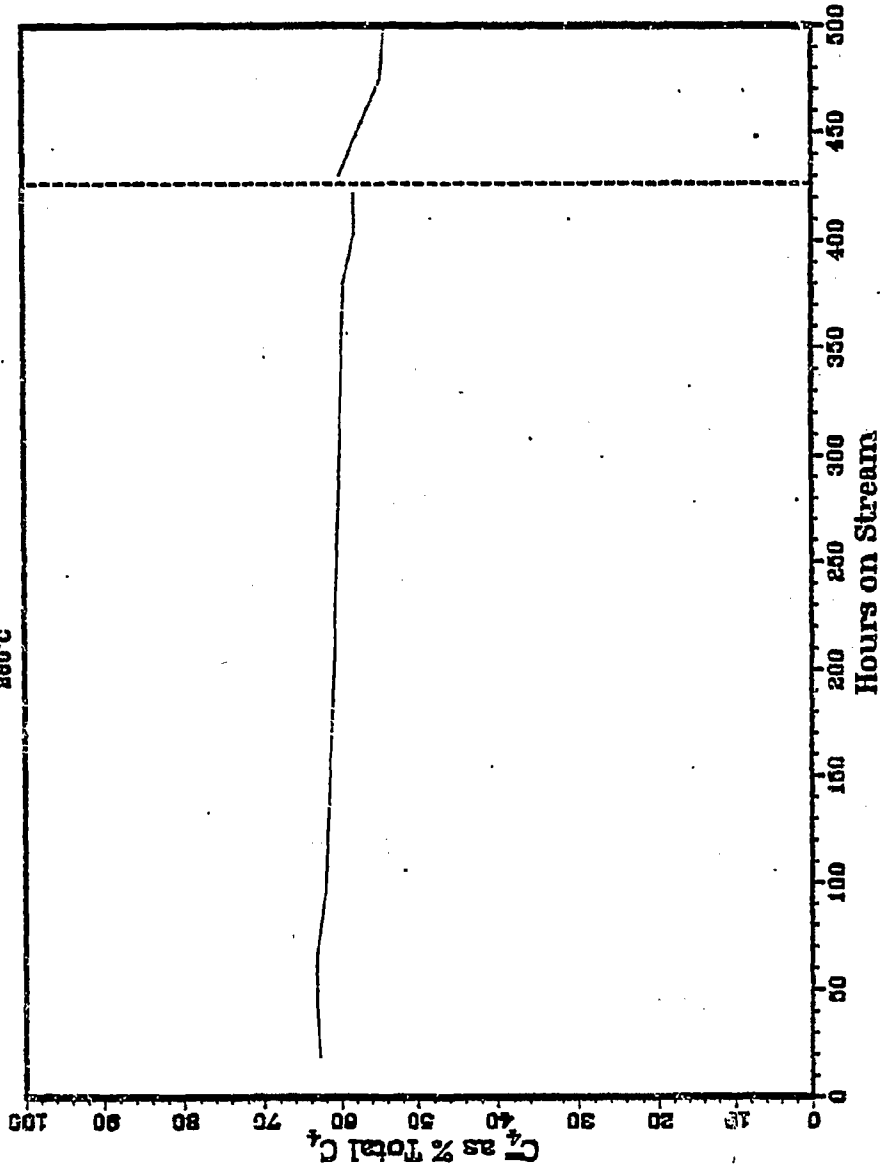


Fig. B56

RUN 12185-07

111 H₂O
500 PSIG
860°C

500 PSIG

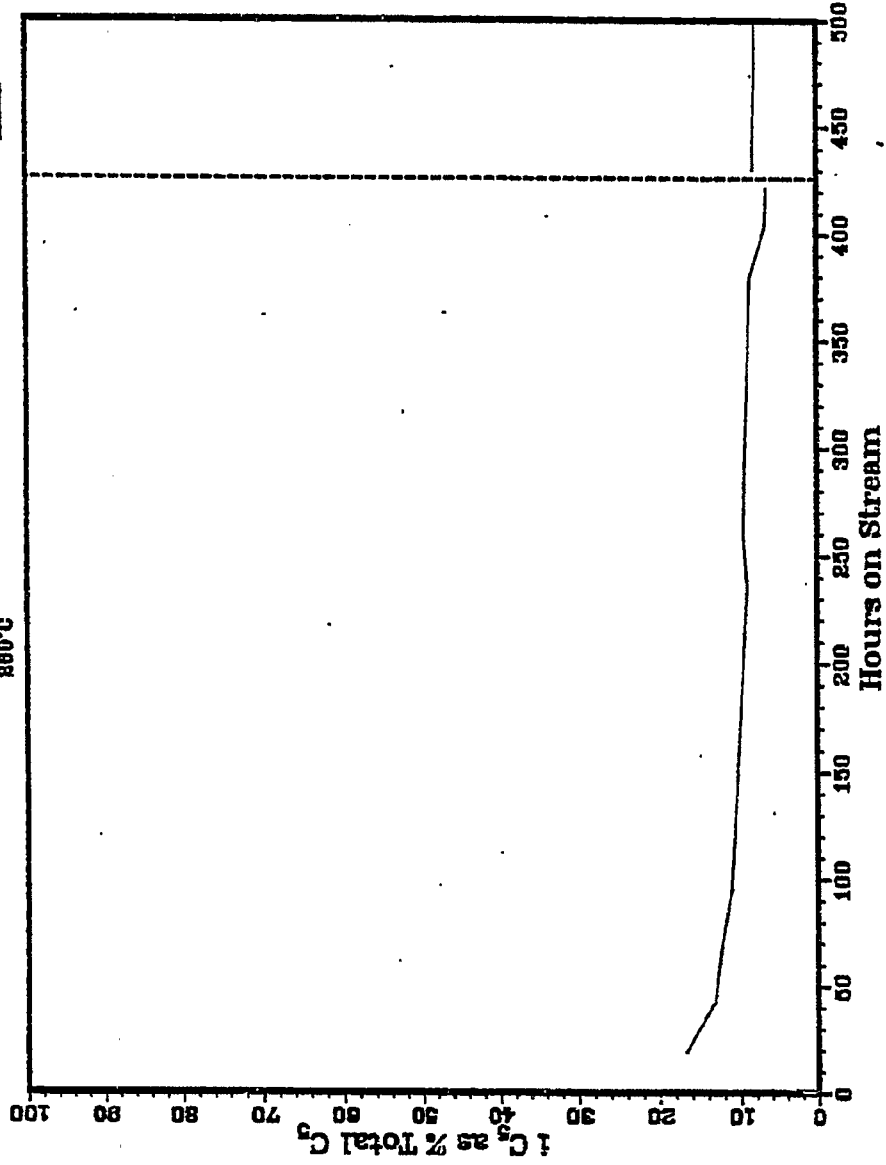


Fig. B57

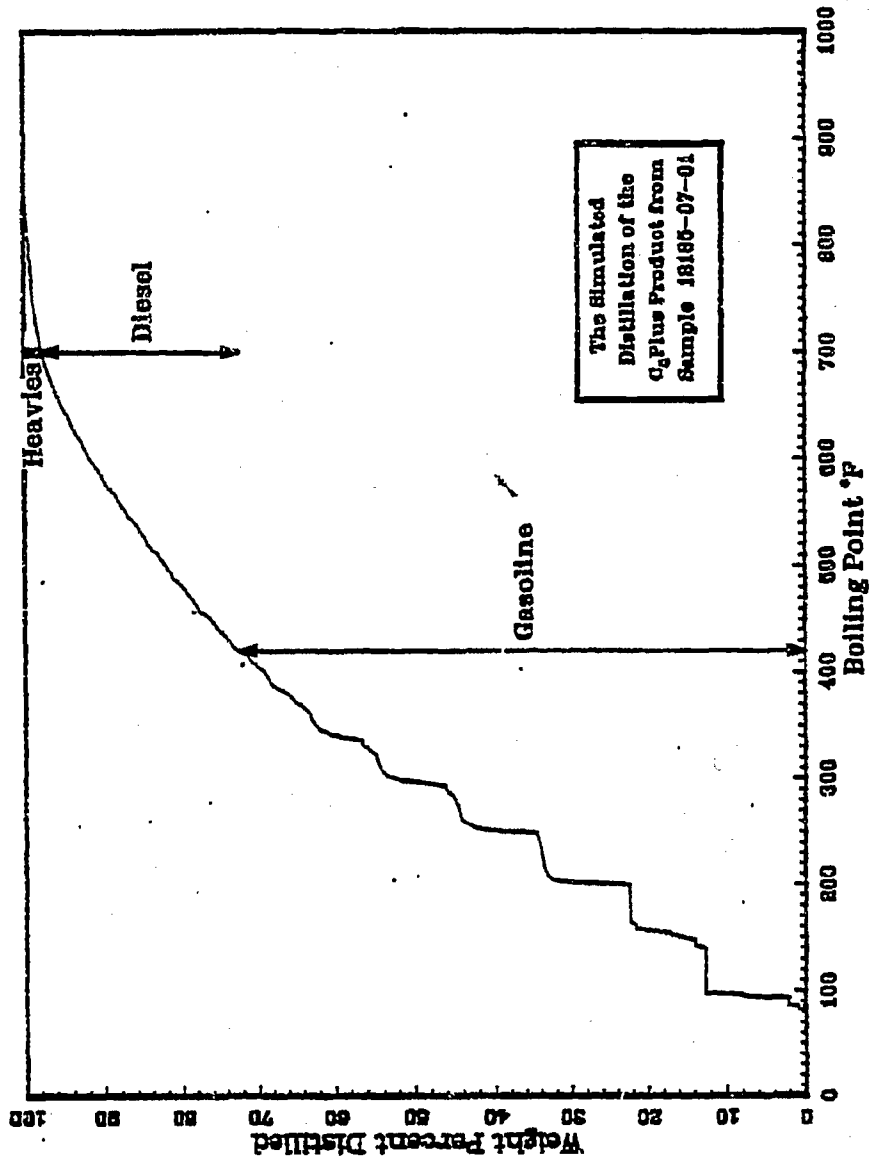


Fig. B58

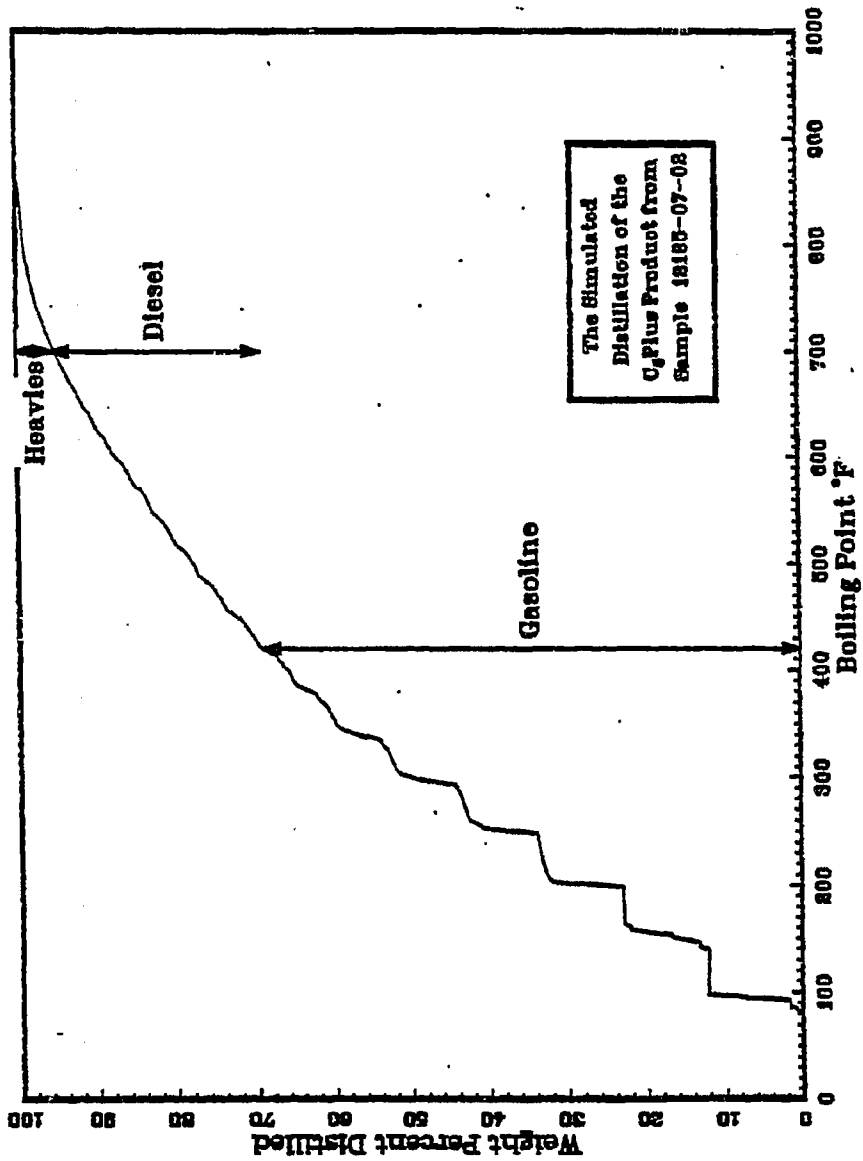


Fig. B59

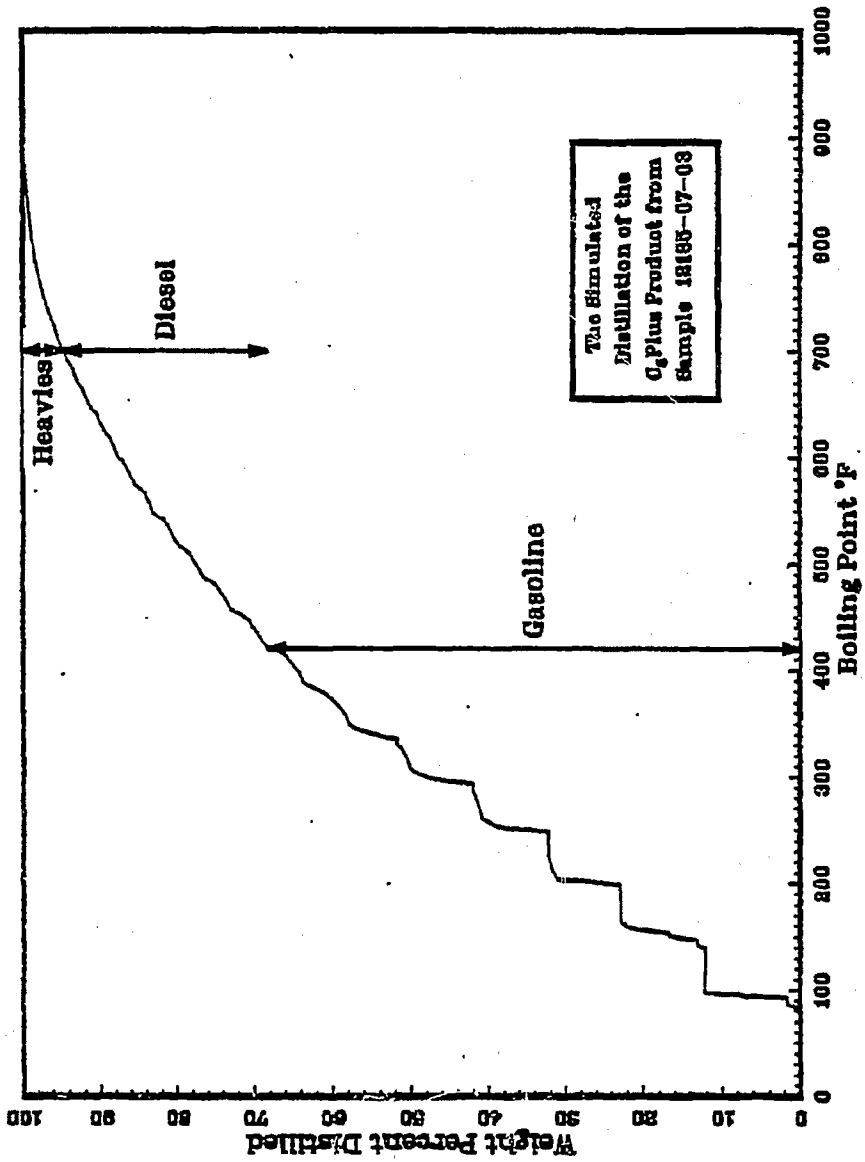


Fig. B60

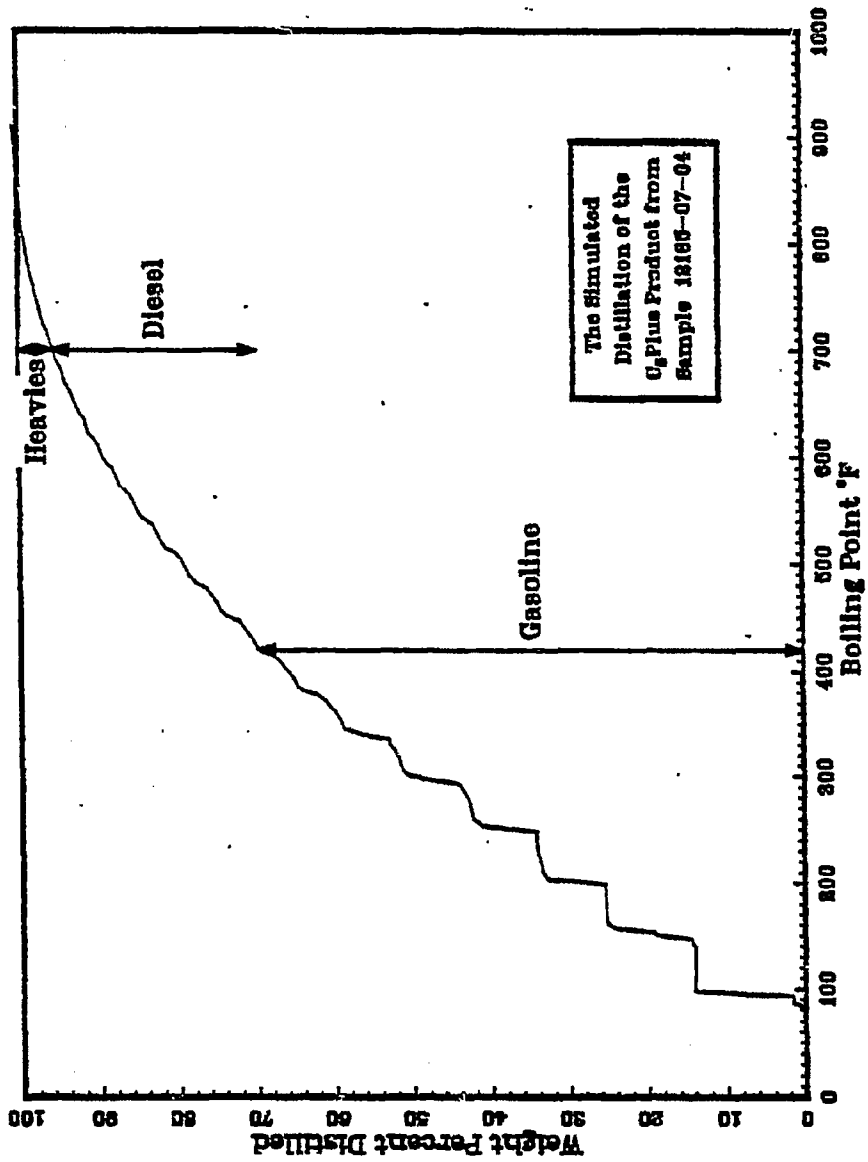


Fig. B61

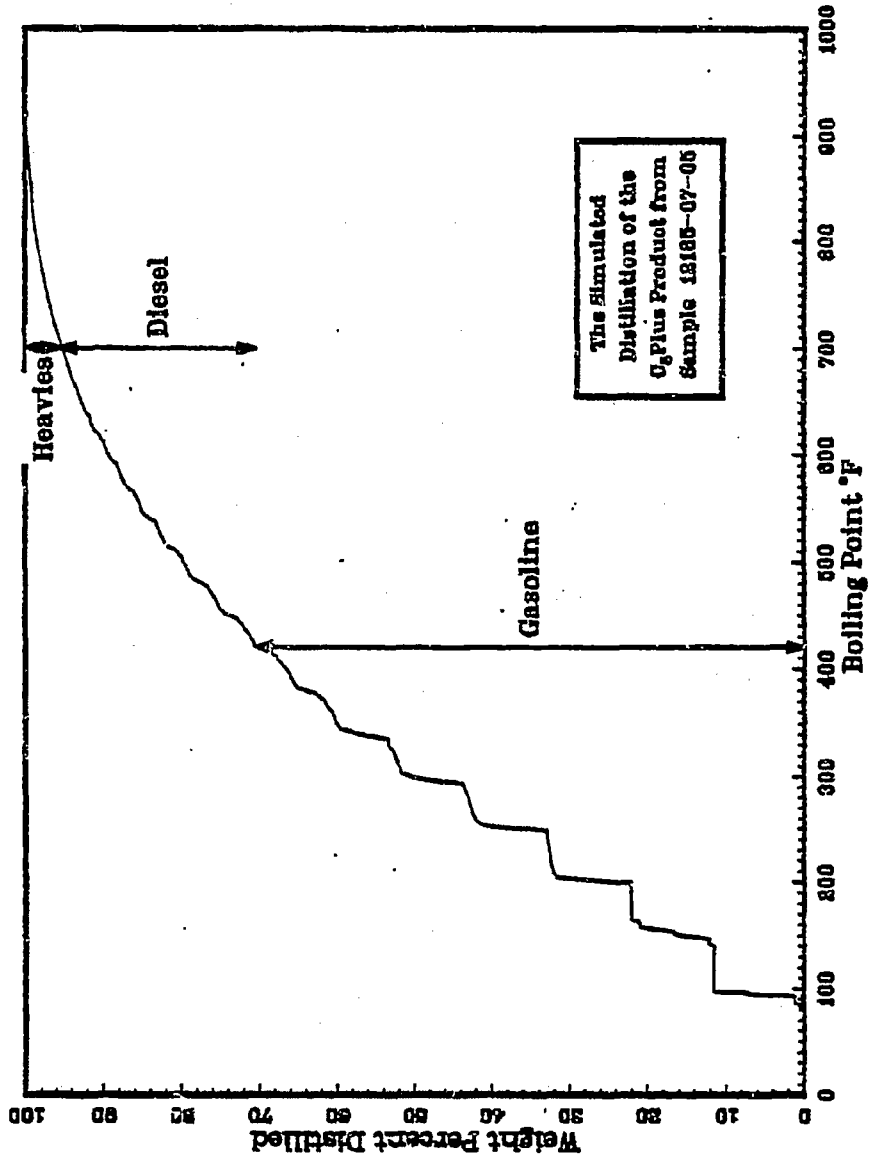


Fig. B62

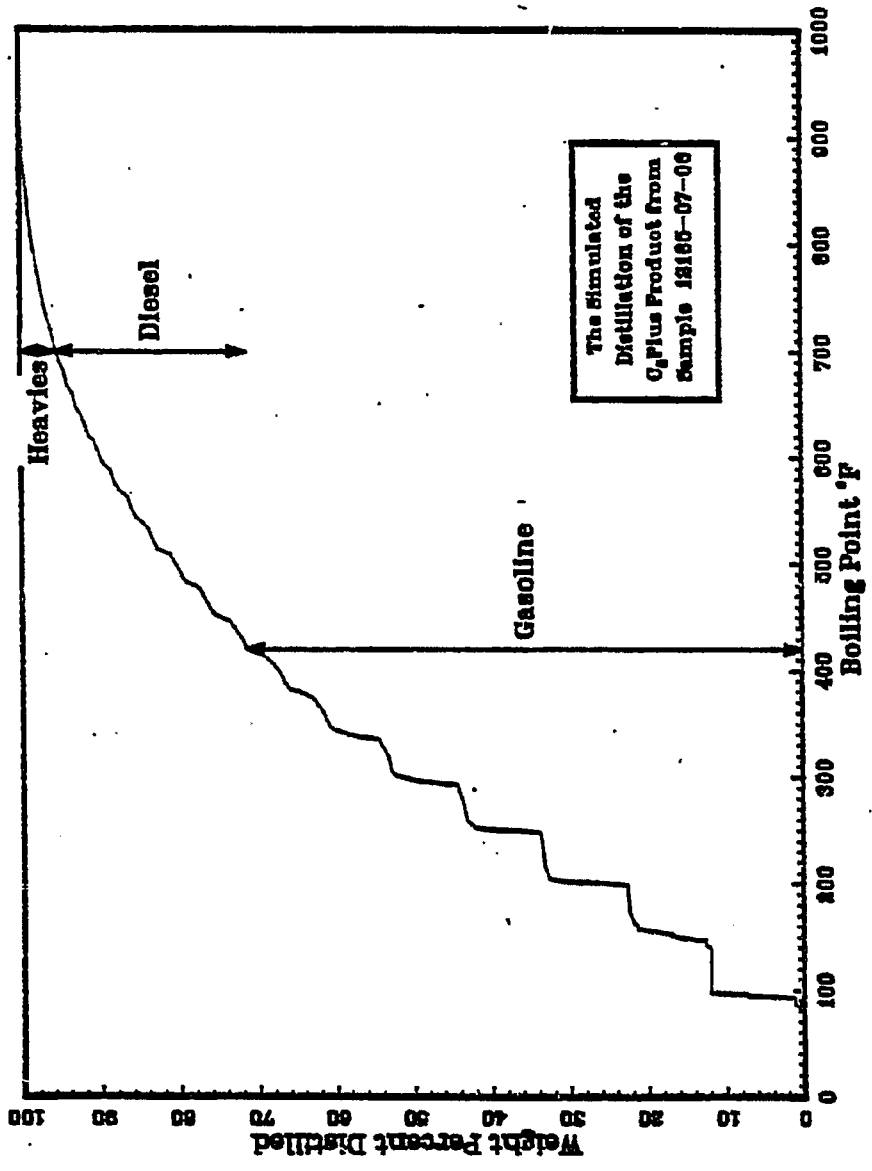


Fig. B63

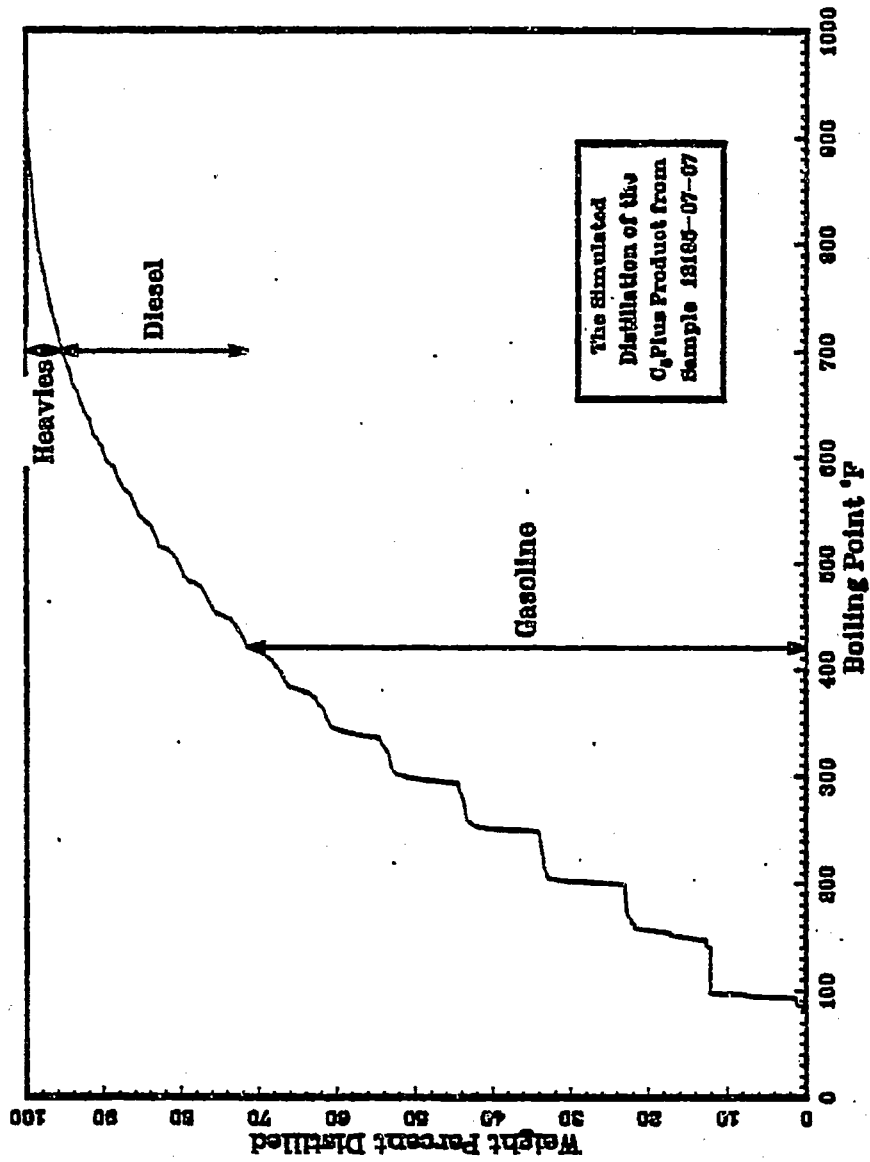


Fig. B64

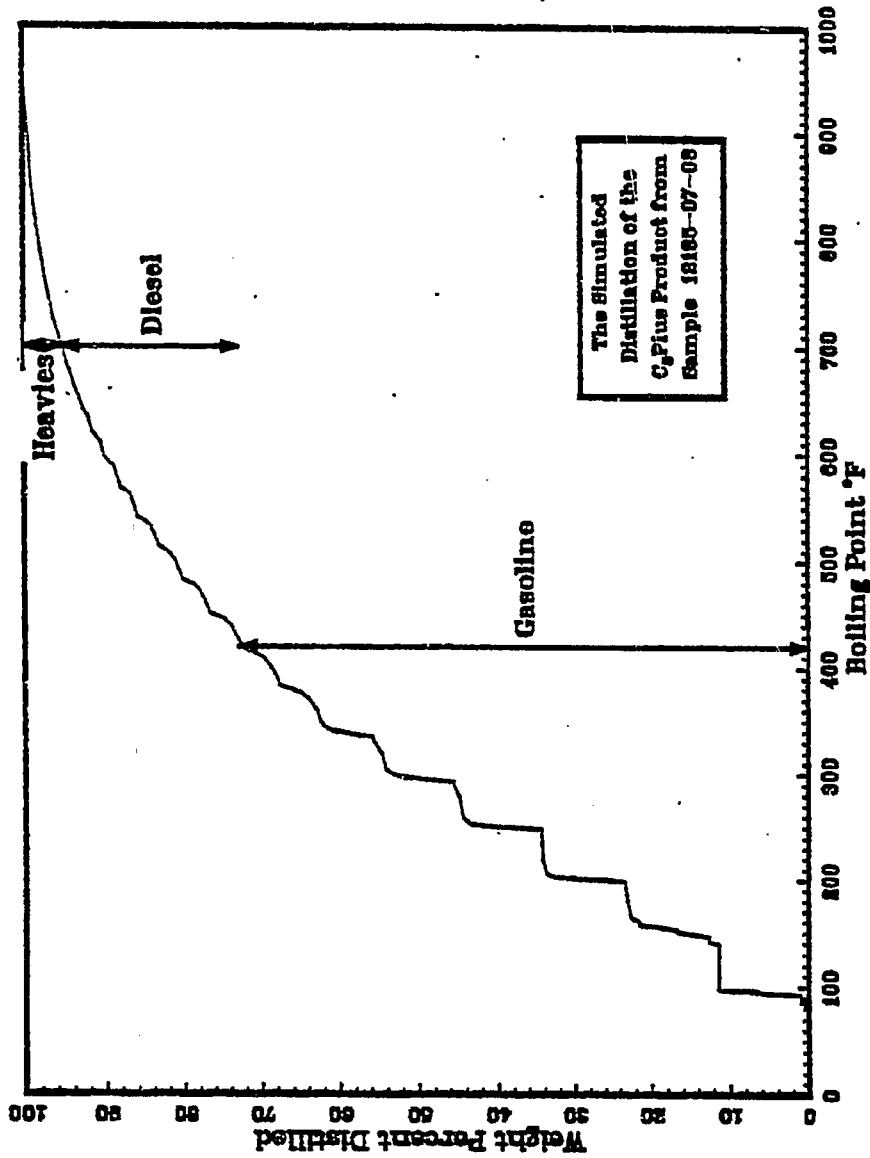


Fig. B65

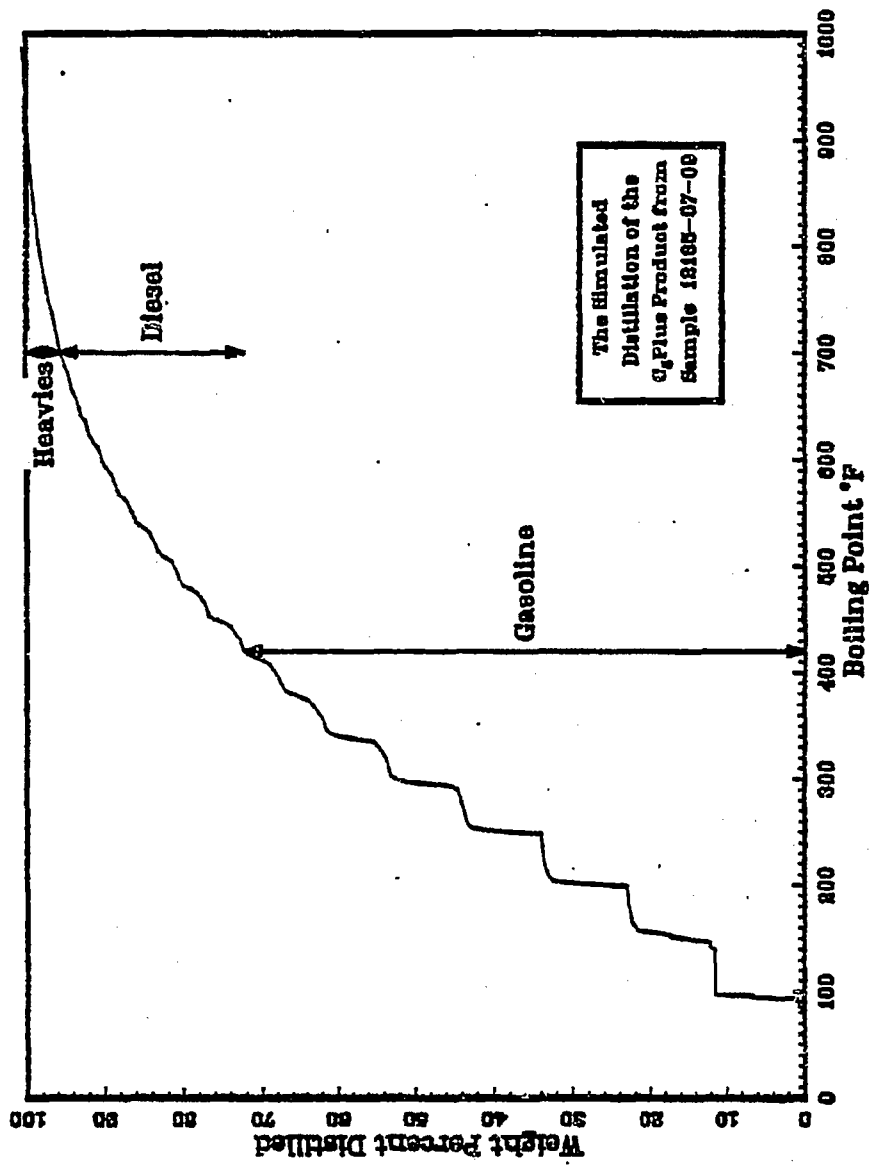


Fig. B66

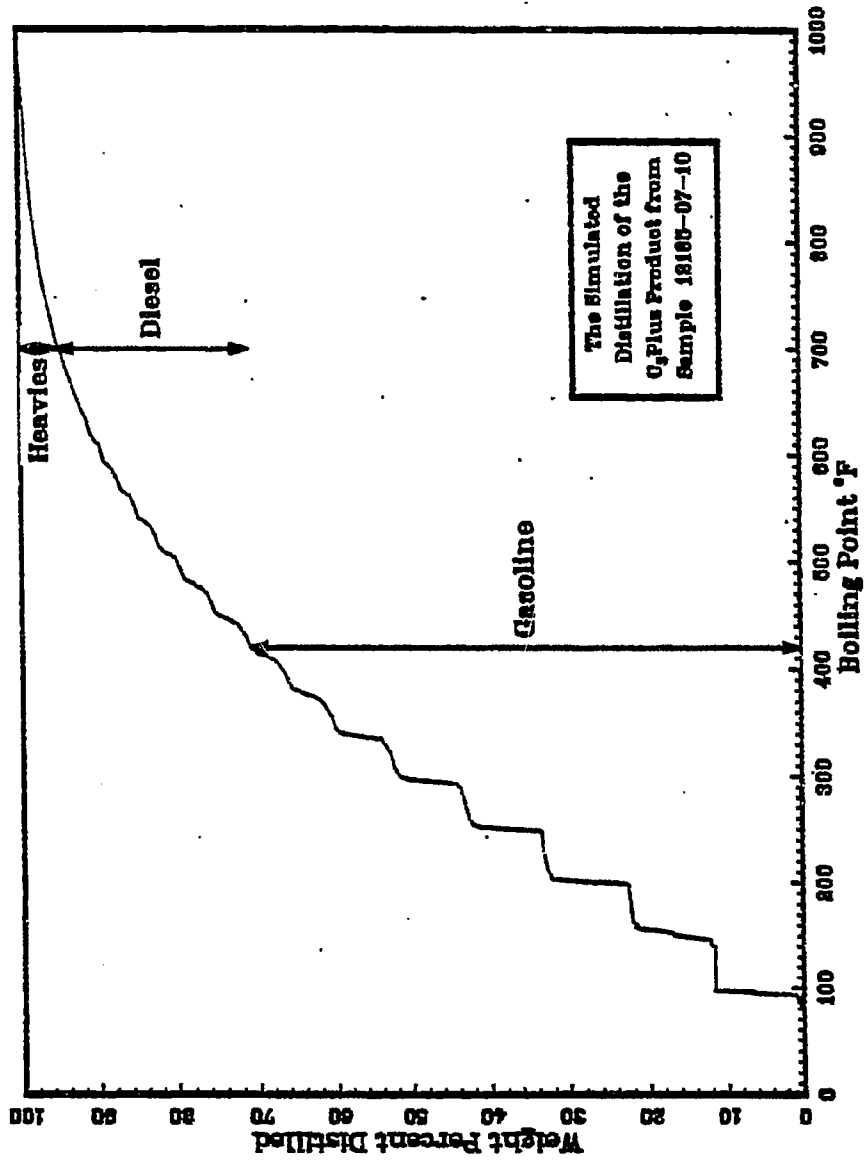


Fig. B67

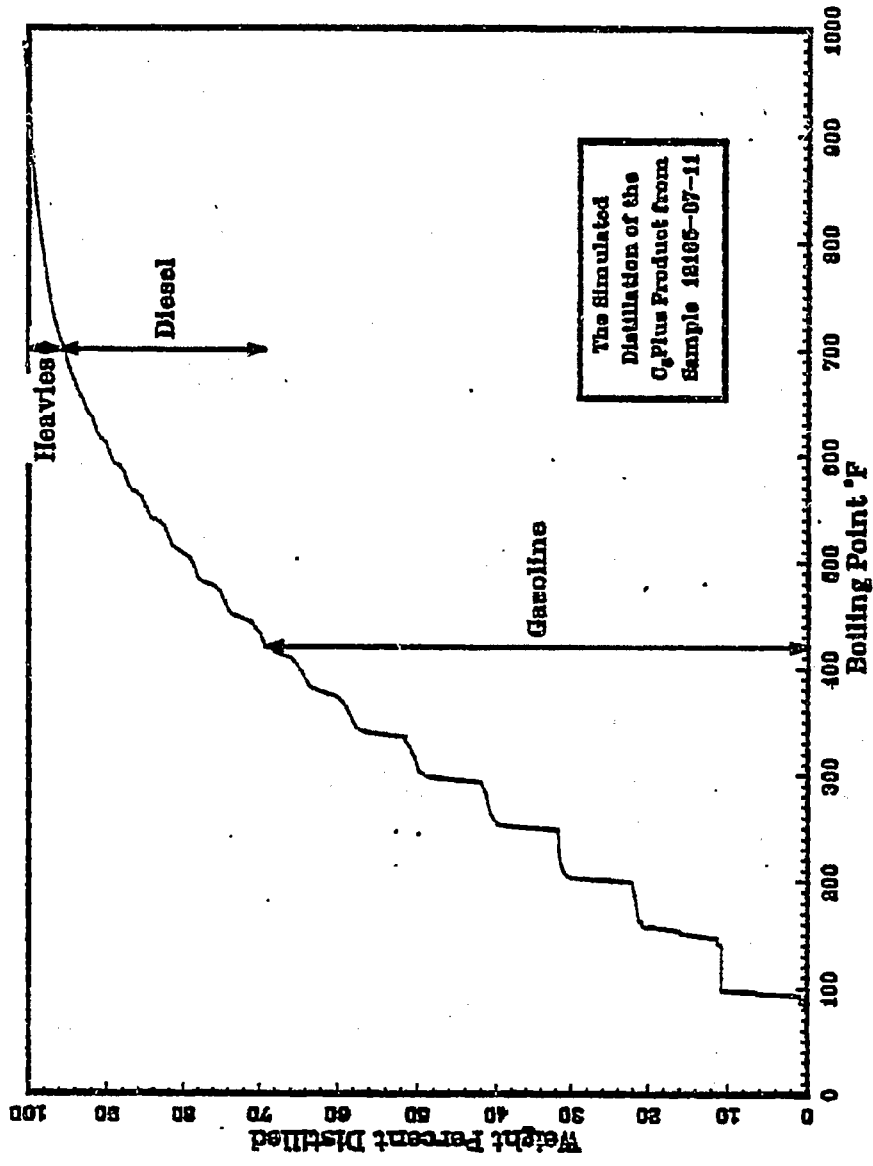


Fig. B68

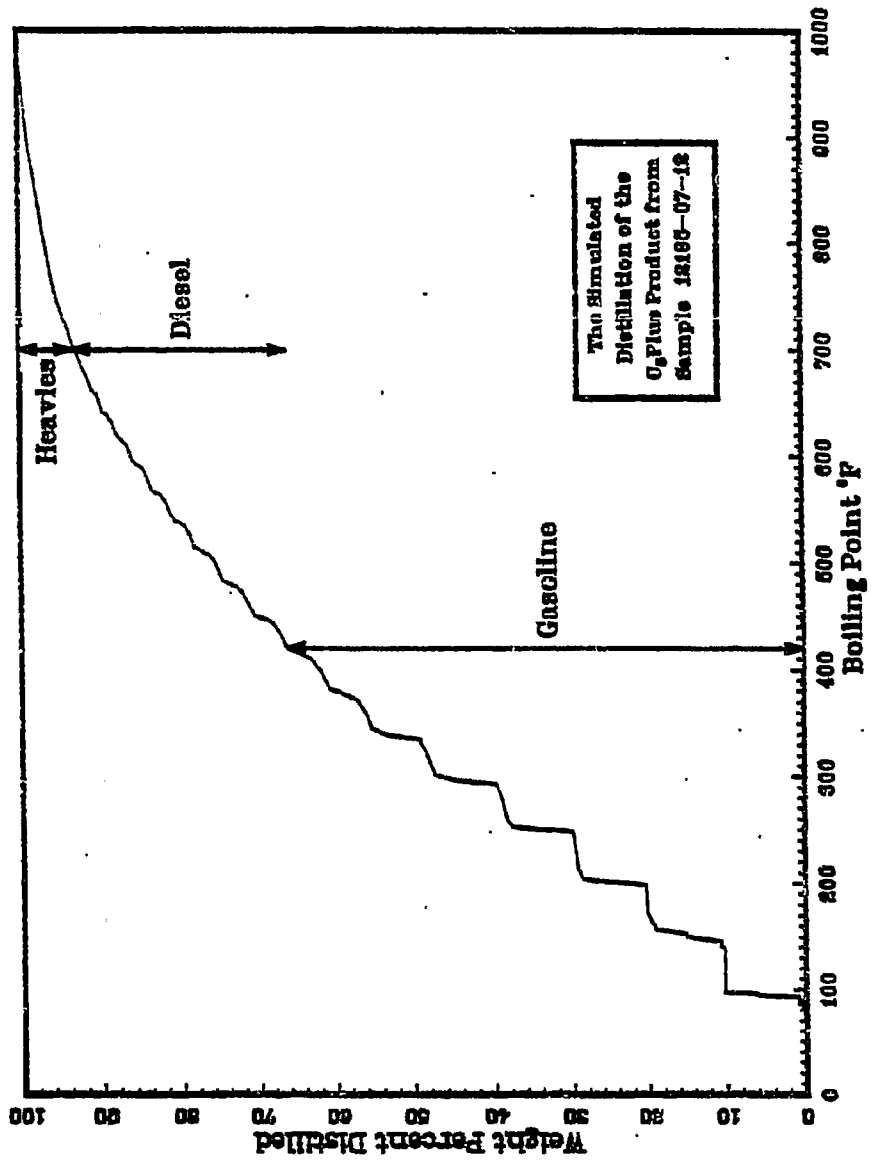


Fig. B69

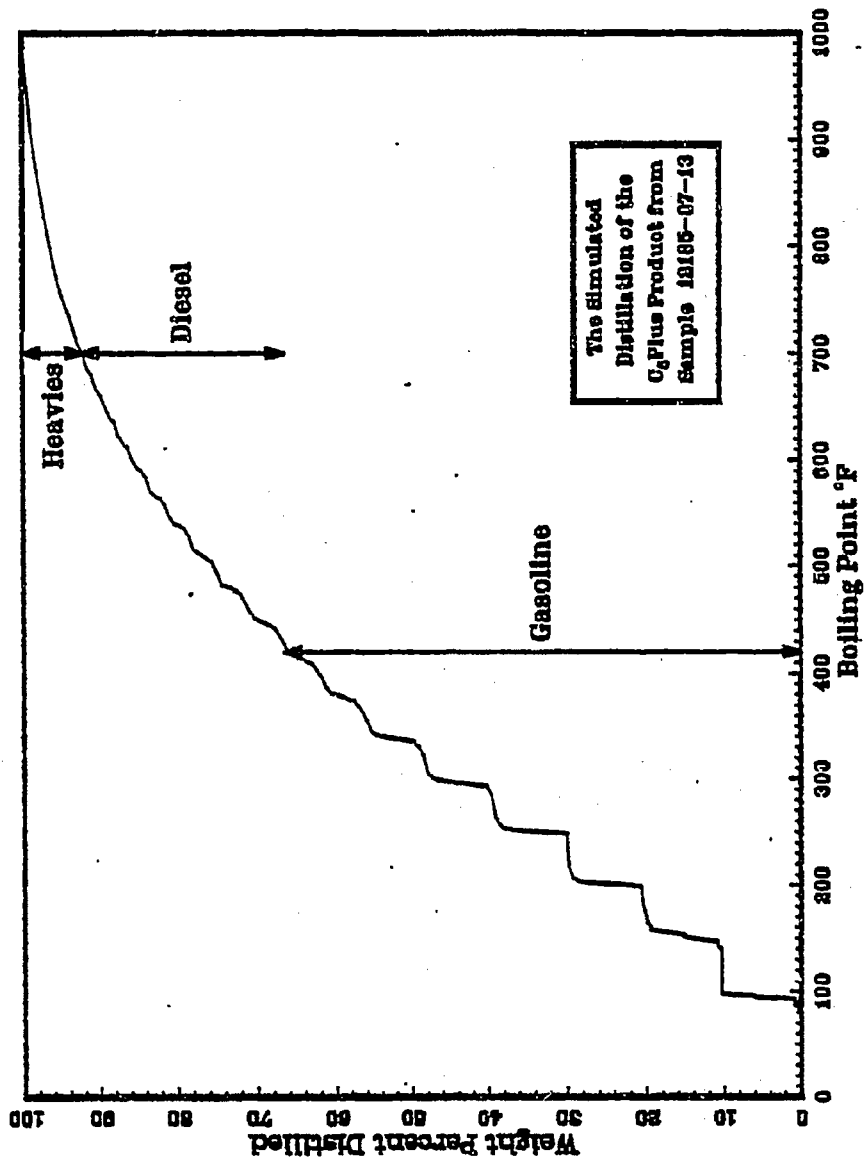


Fig. B70

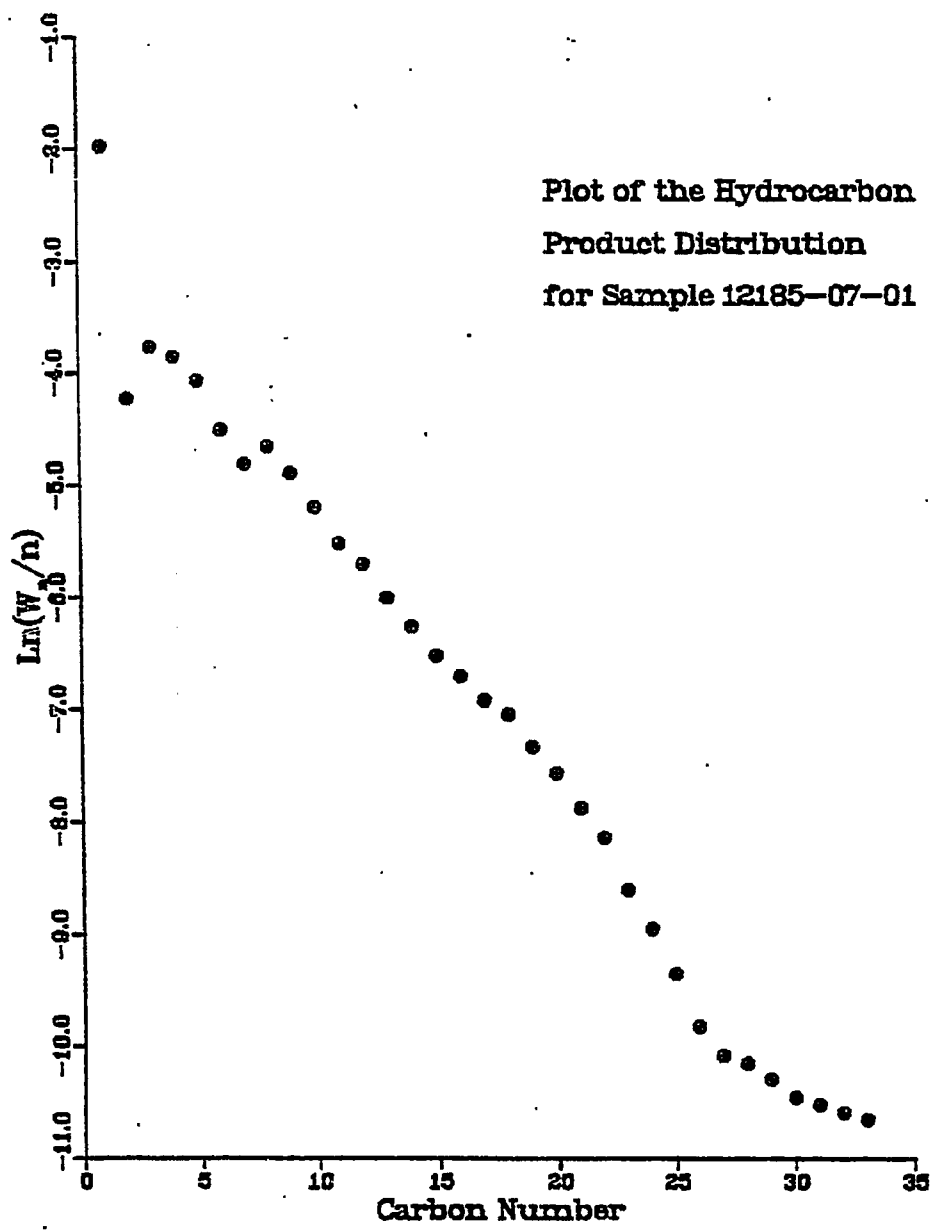


Fig. B71

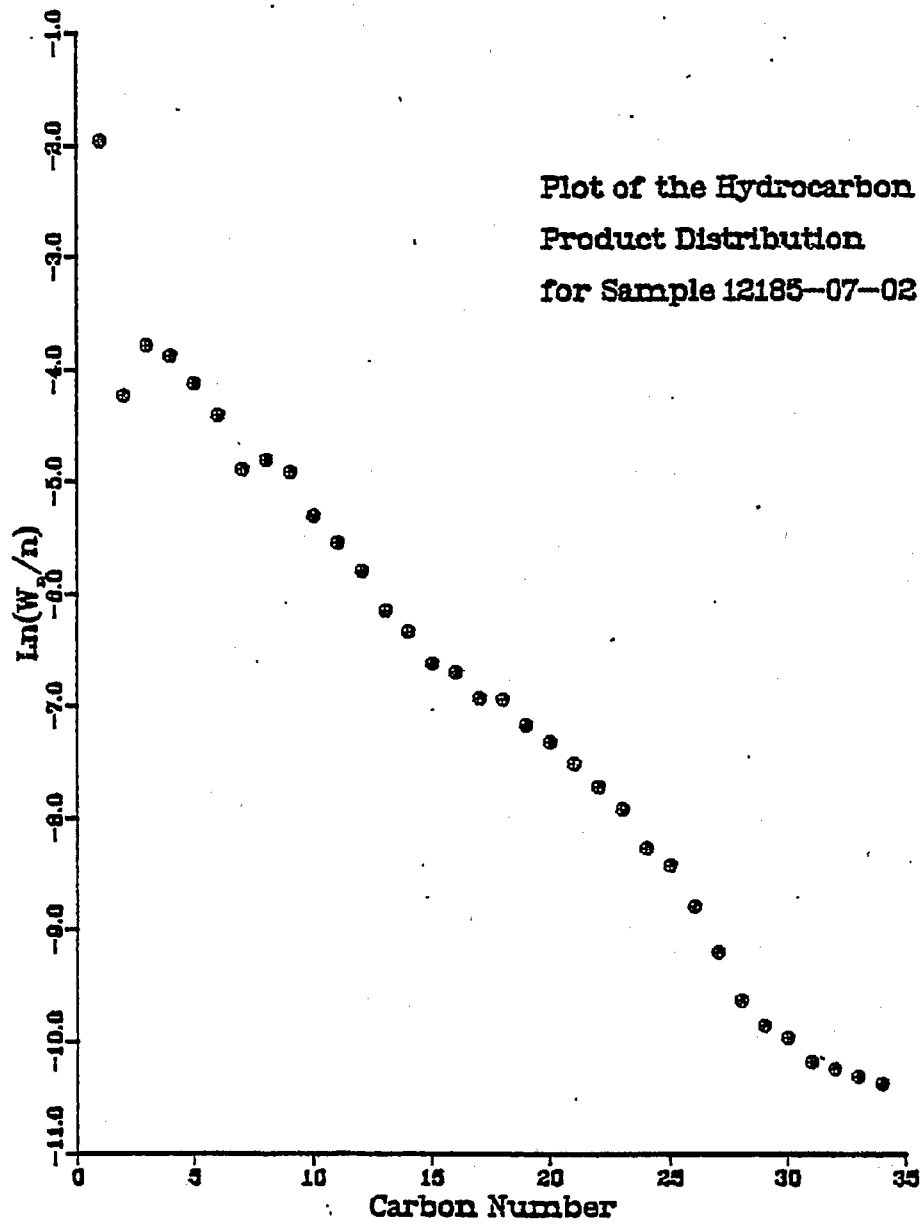


Fig. B72

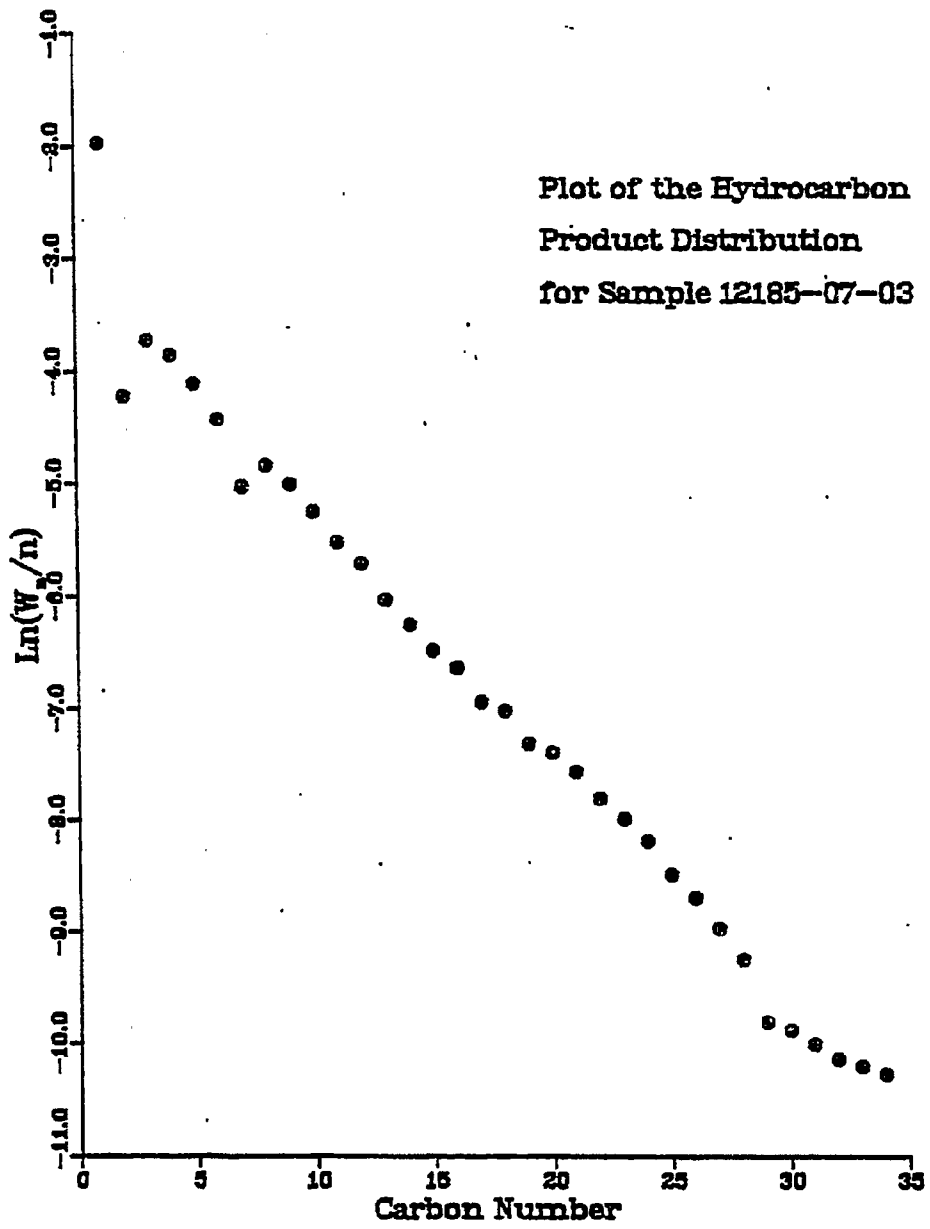


Fig. B73

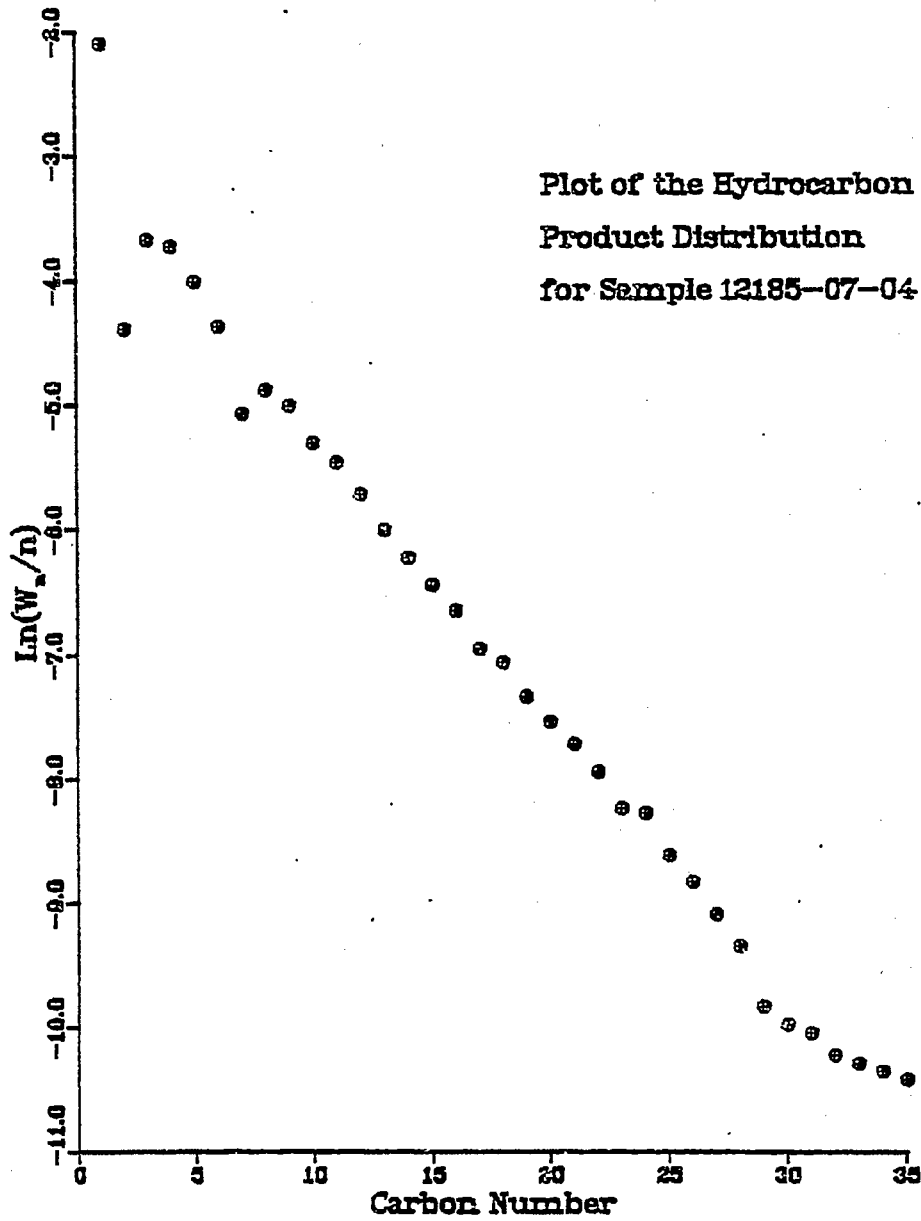


Fig. B74

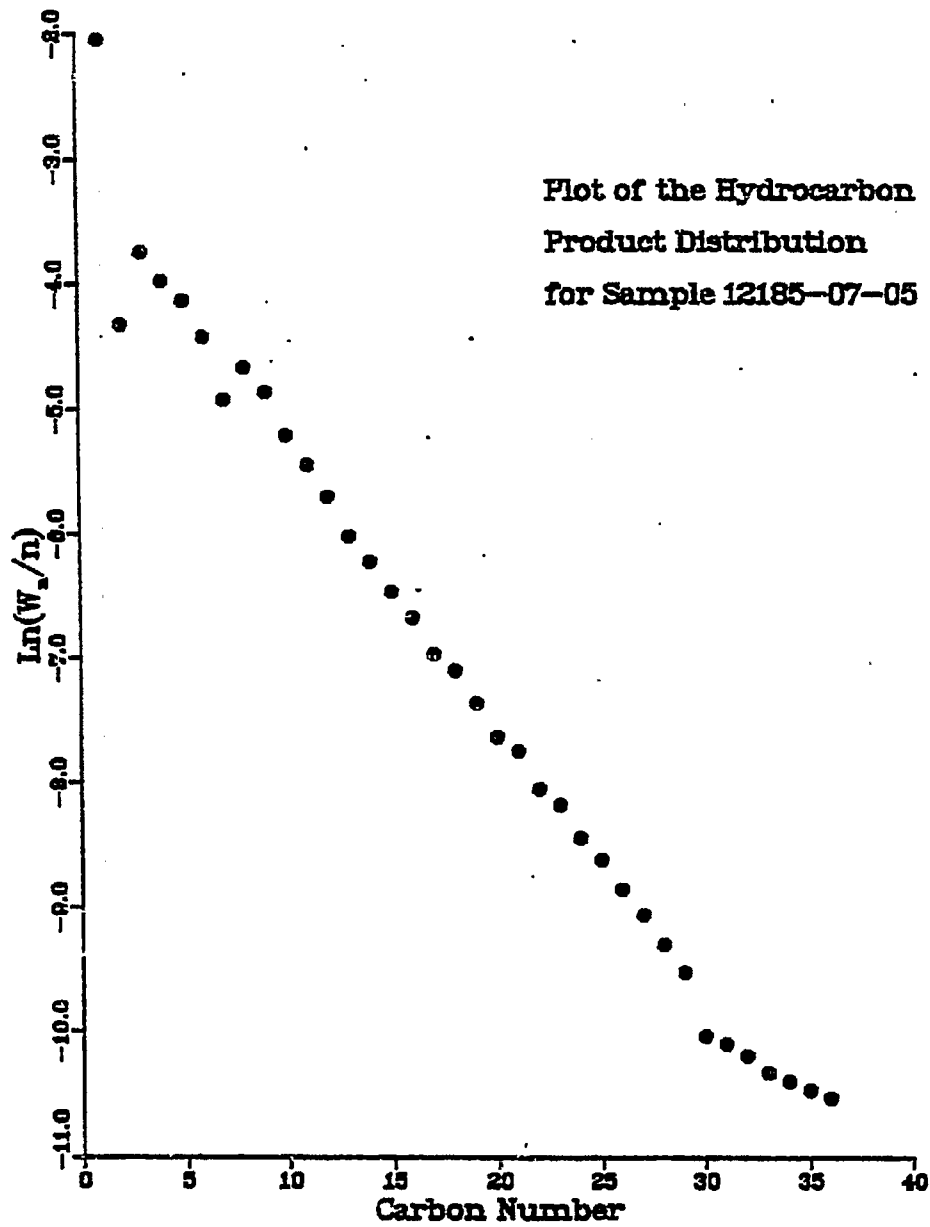


Fig. B75

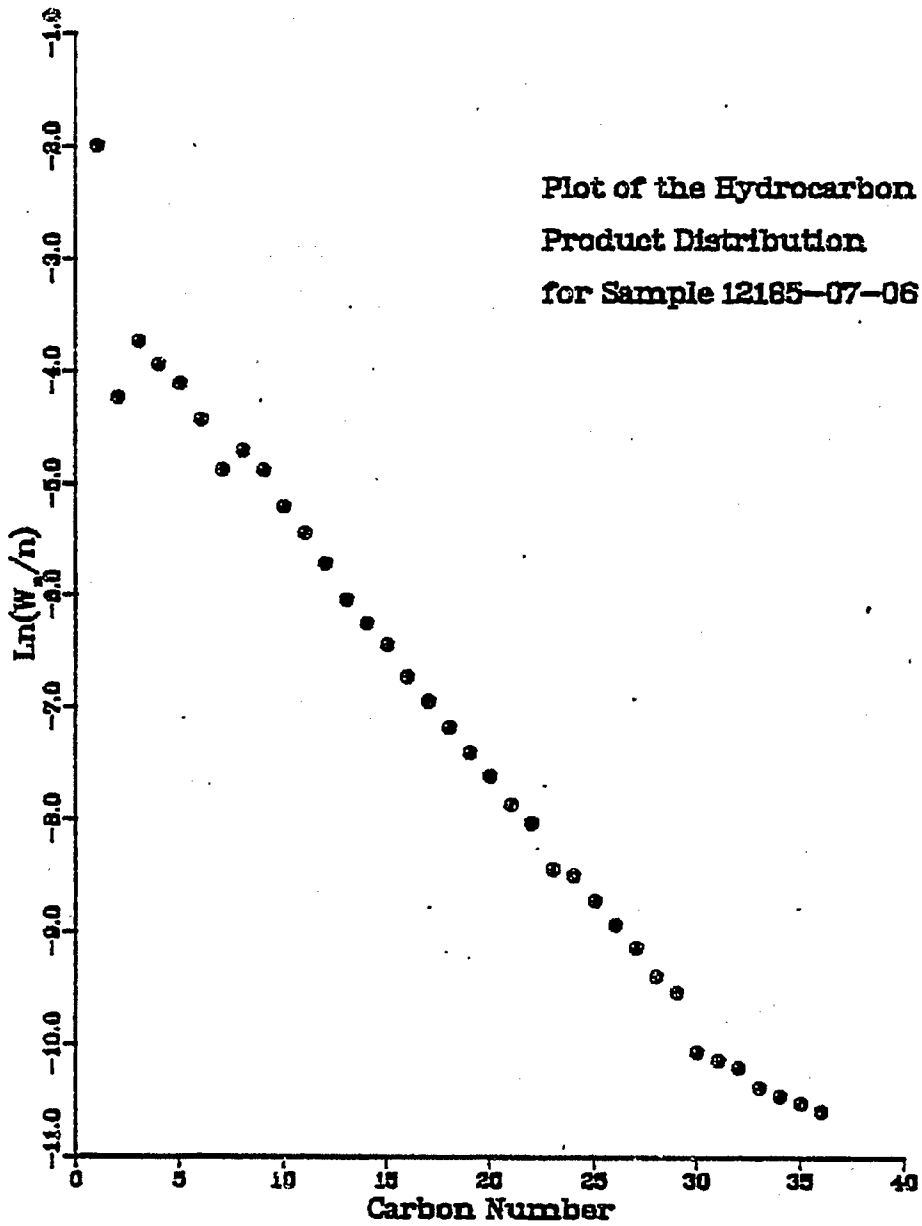


Fig. B76

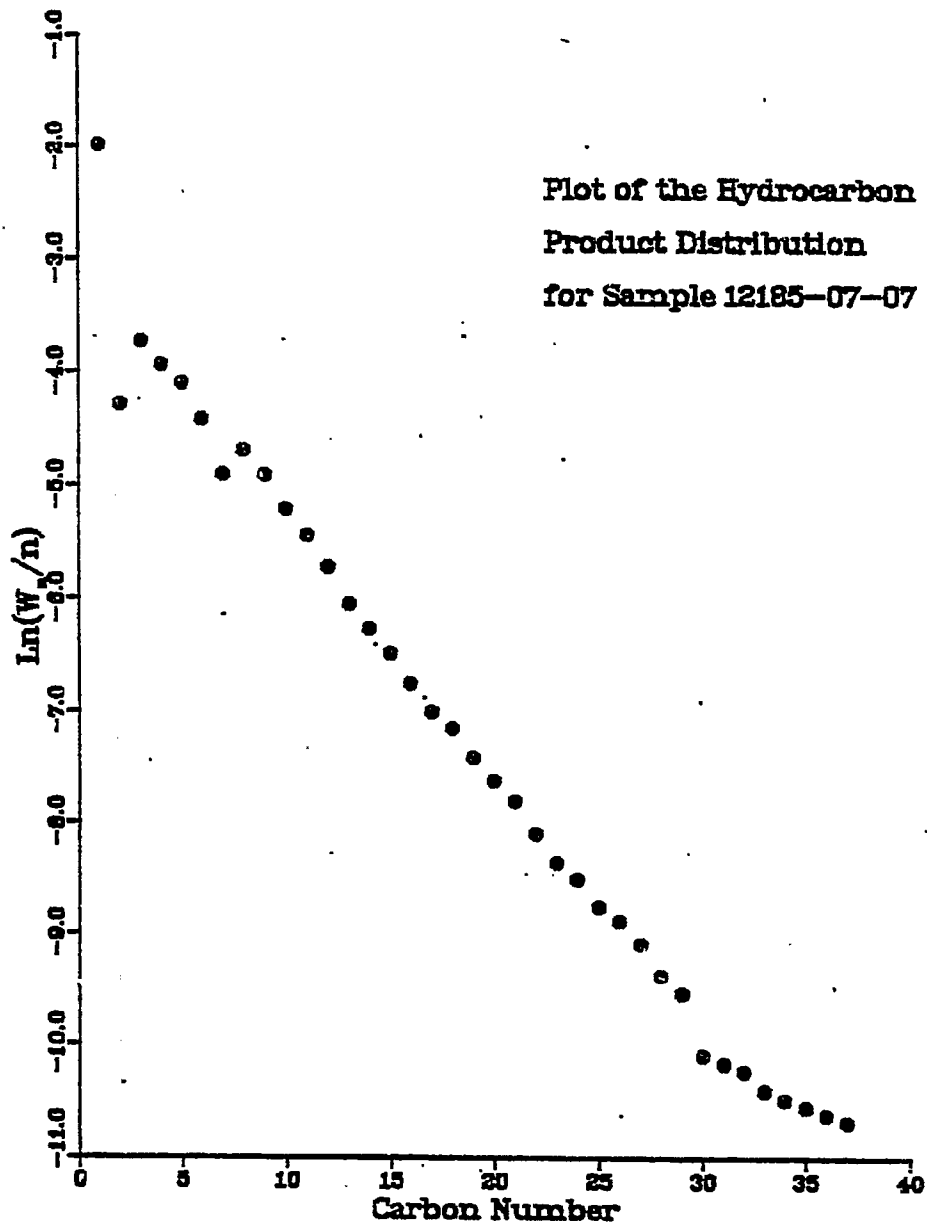


Fig. B77

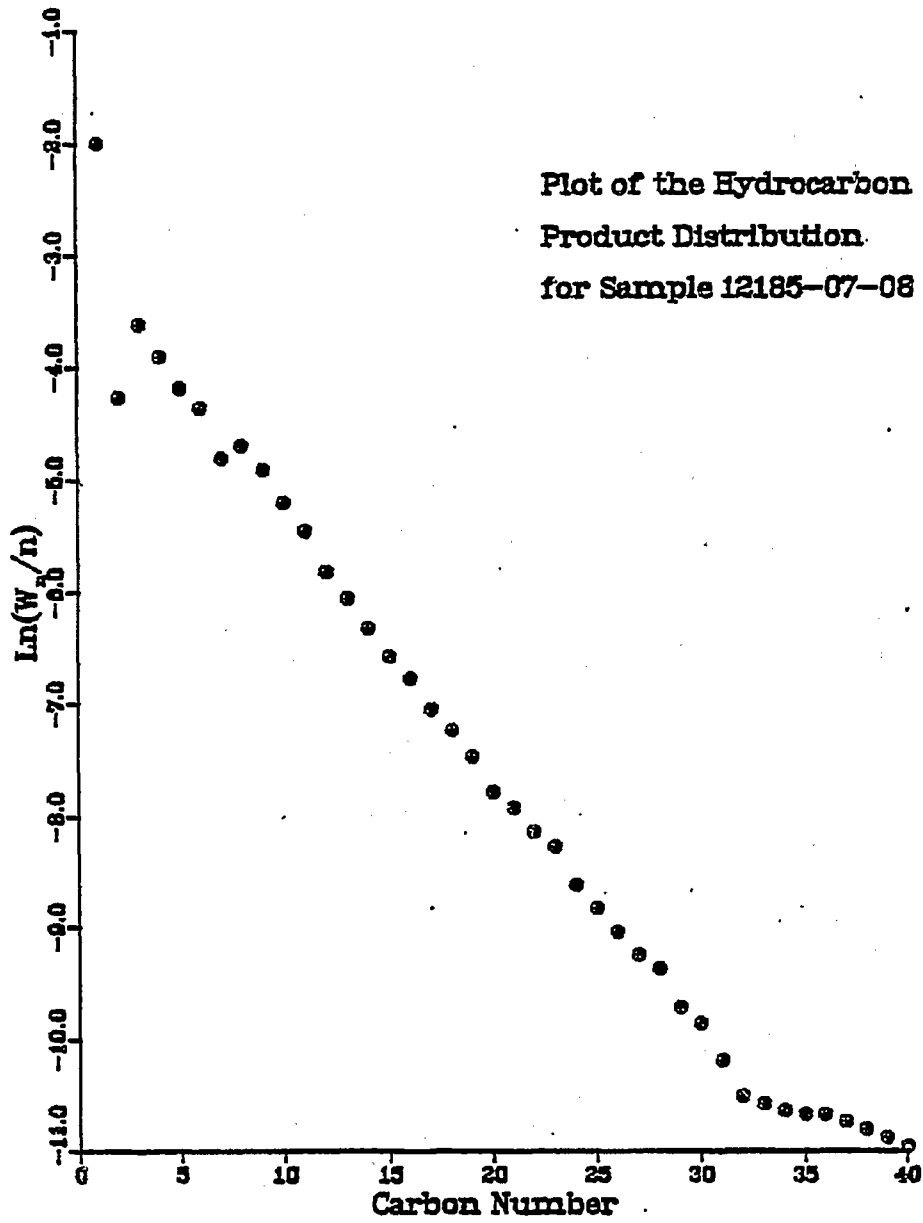


Fig. B78

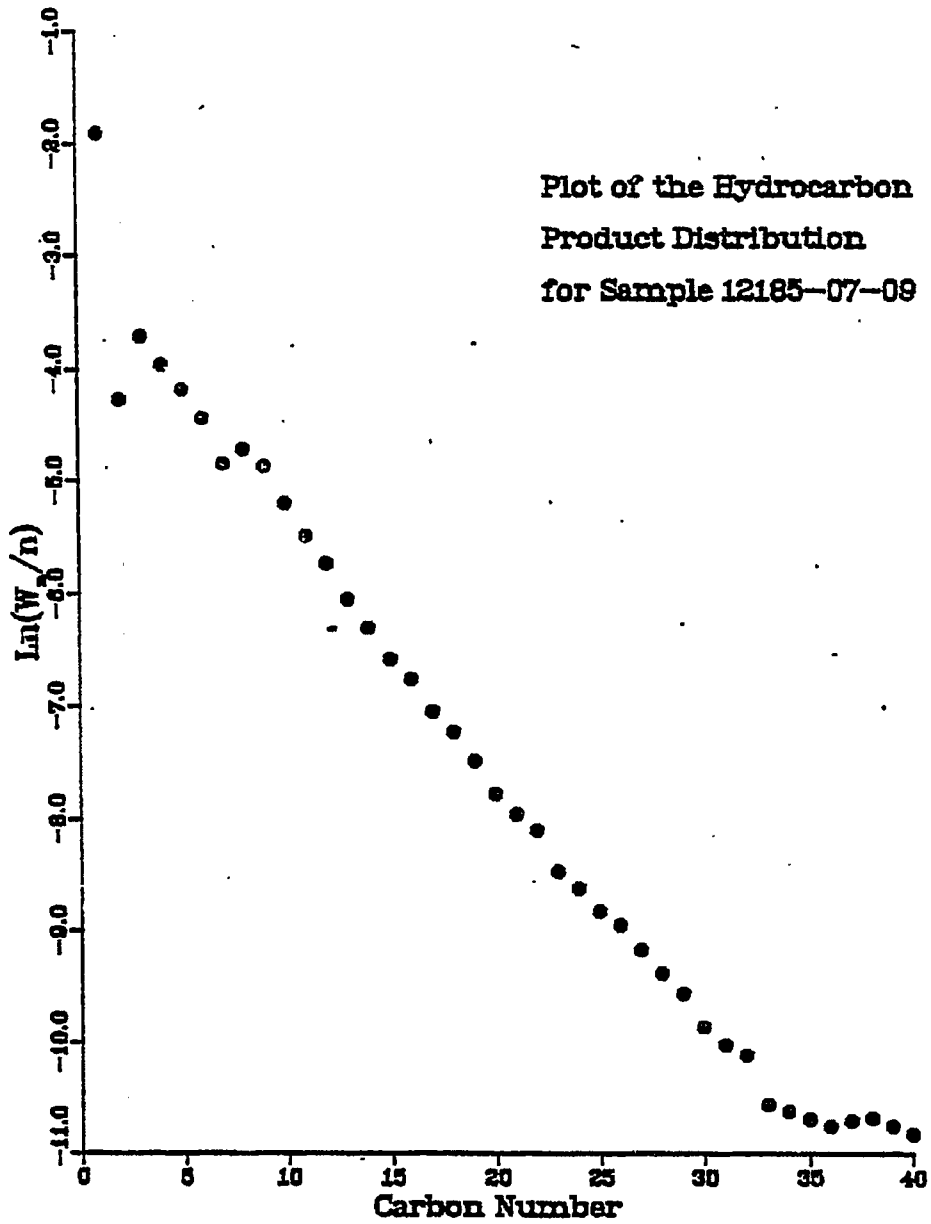


Fig. B79

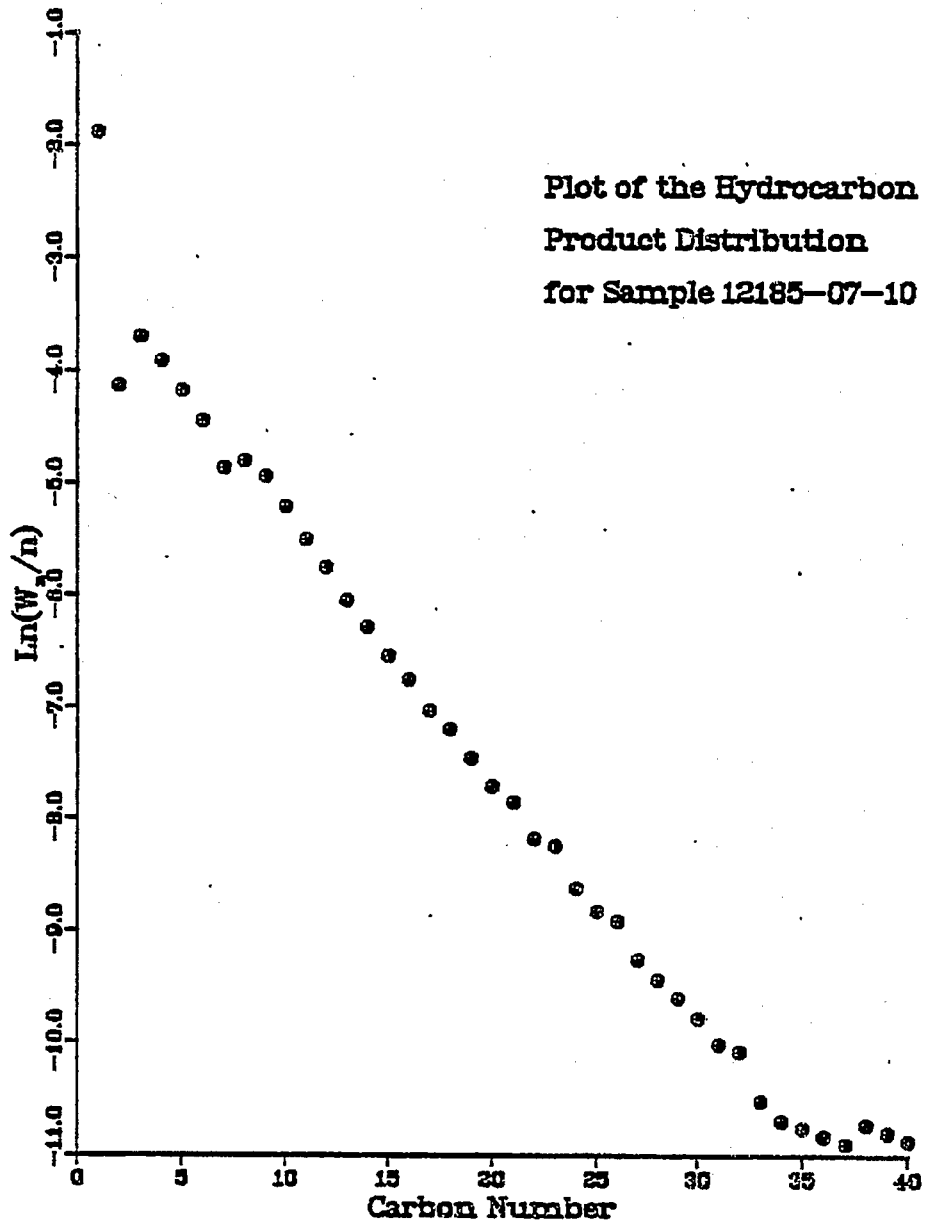


Fig. B80

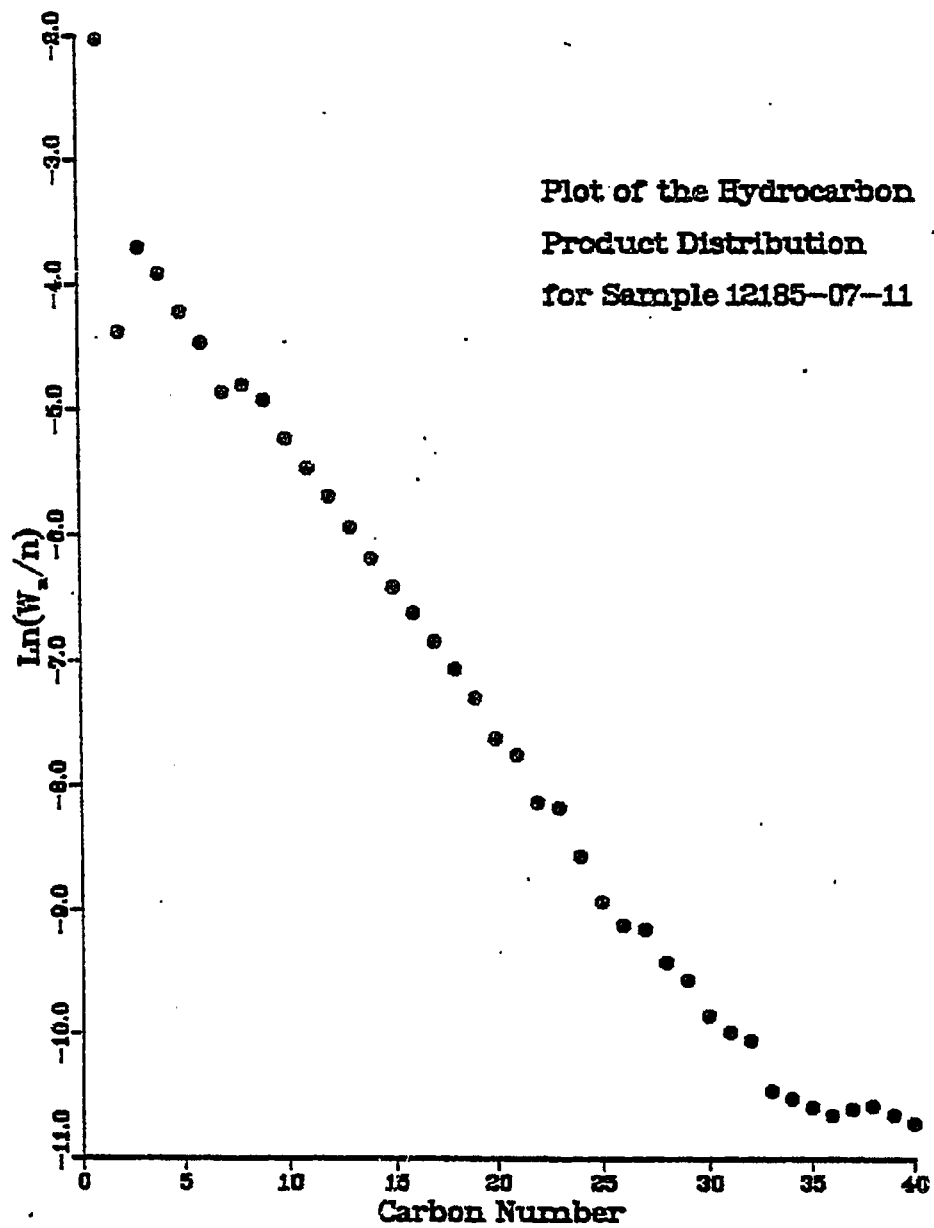


Fig. B81

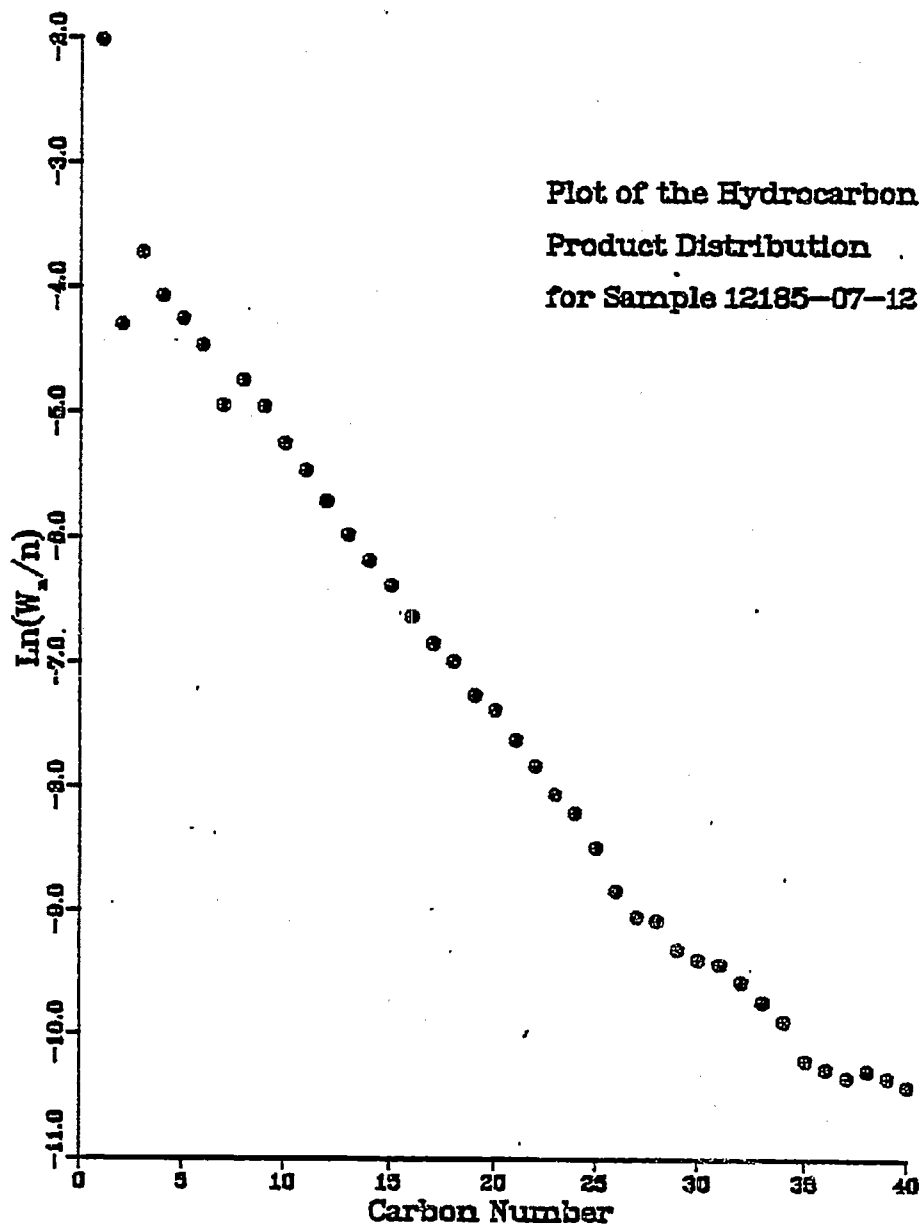


Fig. B82

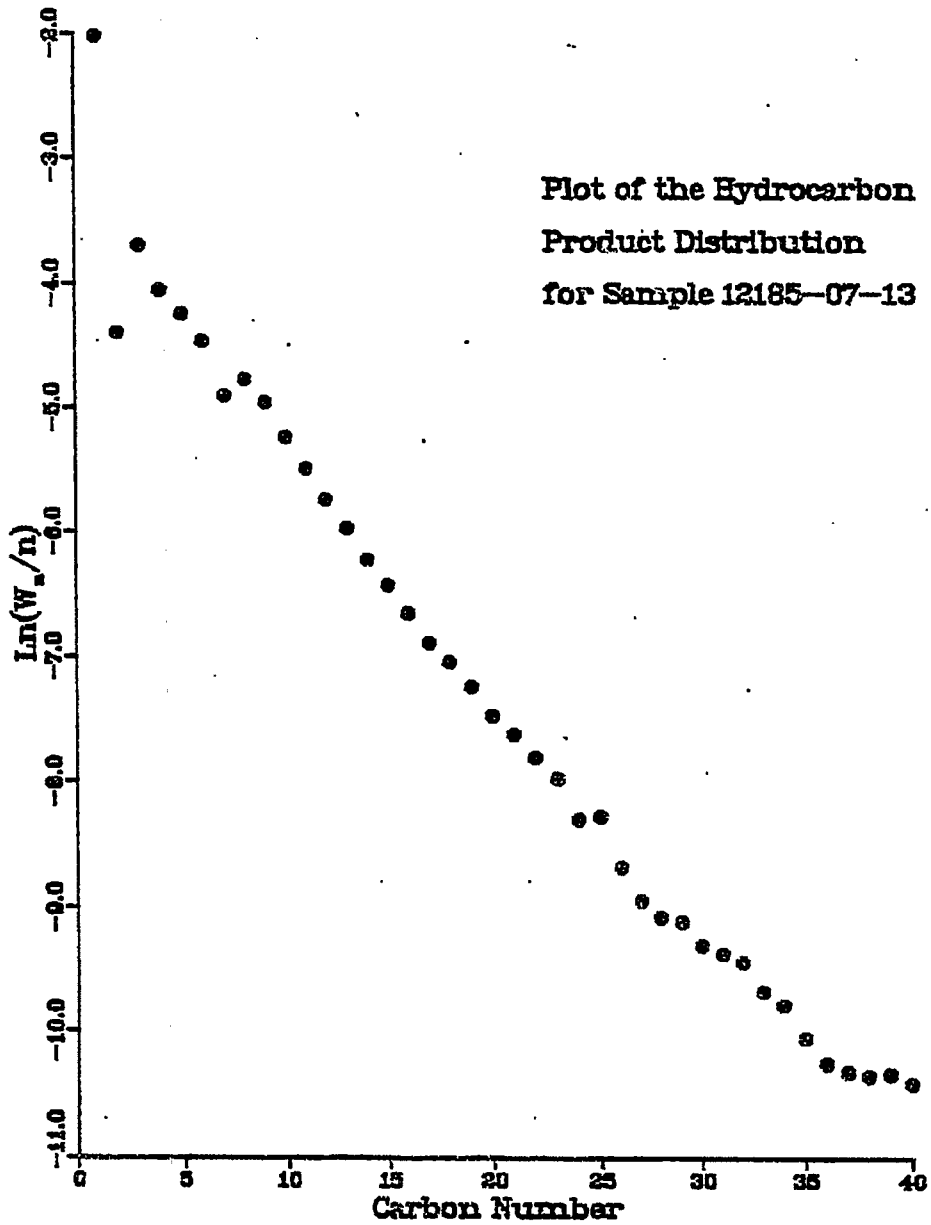


Fig. B83

12185-07-01

12185-07-01

12185-07-01

12185-07-01

12185-07-01

12185-07-01

12185-07-01

12185-07-01

12185-07-01

Fig. B84

- B102 -

407

OVER TEMP AT 100

SET POINT=275°C

OVER TEMP



SET POINT=275°C SETPOINT=275°C LIMIT=405°C

SET POINT=350°C SETPOINT=350°C LIMIT=405°C

END

12185-D7-02
12185-7-2

Fig. B85

OVEN TEMP NOT READING

103

SET: 11000 1.20

OVEN

TEMP=275°C

OVEN TEMP=275°C SETPT=275°C LIMIT=400°C

OVEN TEMP=320°C SETPT=320°C LIMIT=400°C

SET: 1700 0.1

12185-07-02

12185-07-02

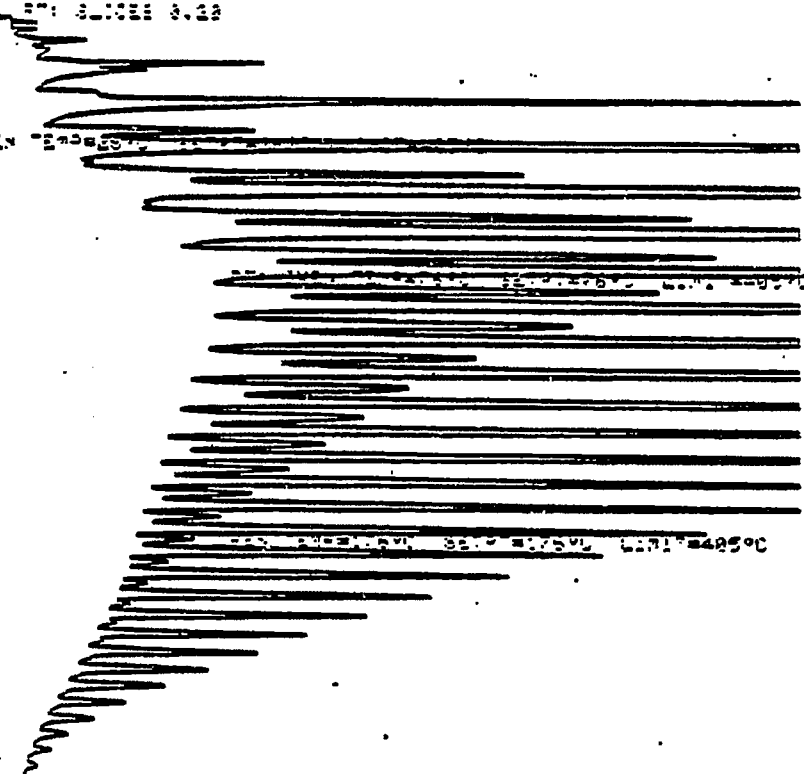
Fig. B86

OVEN TEMP NOT READY

TCT

RT: 3.1000 3.20

RT: OVEN TEMP



RT: 3.1000 3.20 LIMIT=40500

RT: OVEN TEMP=27500 GETTEMP=27500 LIMIT=40500

RT: OVEN TEMP=32000 GETTEMP=32000 LIMIT=40500

RT: 3.1000 3.20

12185-07-04

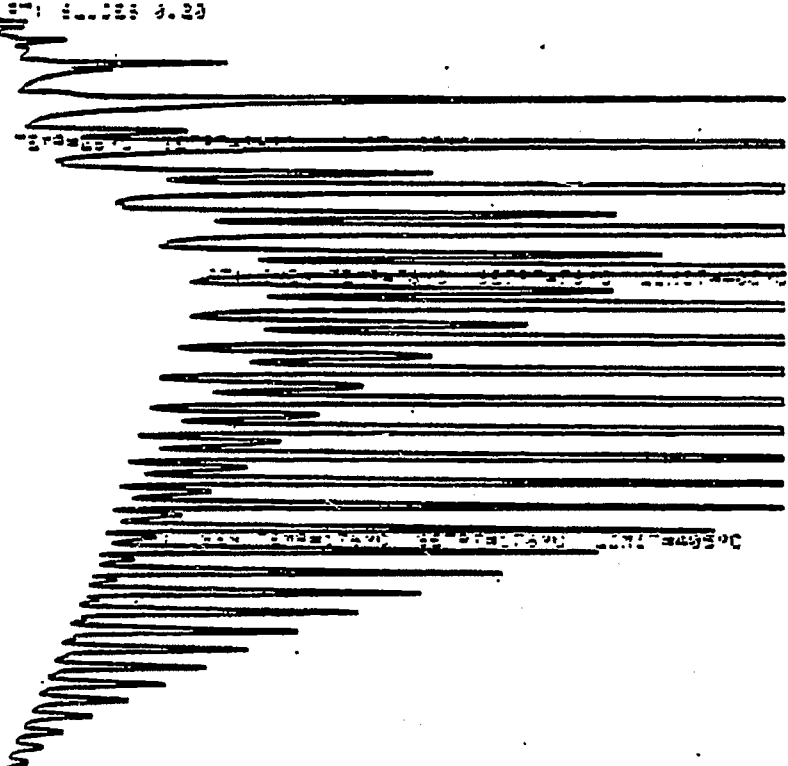
Fig. B87

OVEN TEMP NOT RECD

76T

ST: 11.111 0.23

ST: OVEN



ST: OVEN TEMP=475°C SETPT=475°C LIMIT=400°C

ST: OVEN TEMP=475°C SETPT=475°C LIMIT=400°C

ST: OVEN

12185-07-05

Fig. B88

CCT

OVEN TEMP NOT READ

RT: 21128 9.32

RT: OVEN TEMP



RT: OVEN 2700276°C SETPT=276°C LIMIT=405°C

RT: OVEN 2700276°C SETPT=276°C LIMIT=405°C

12185-07-06

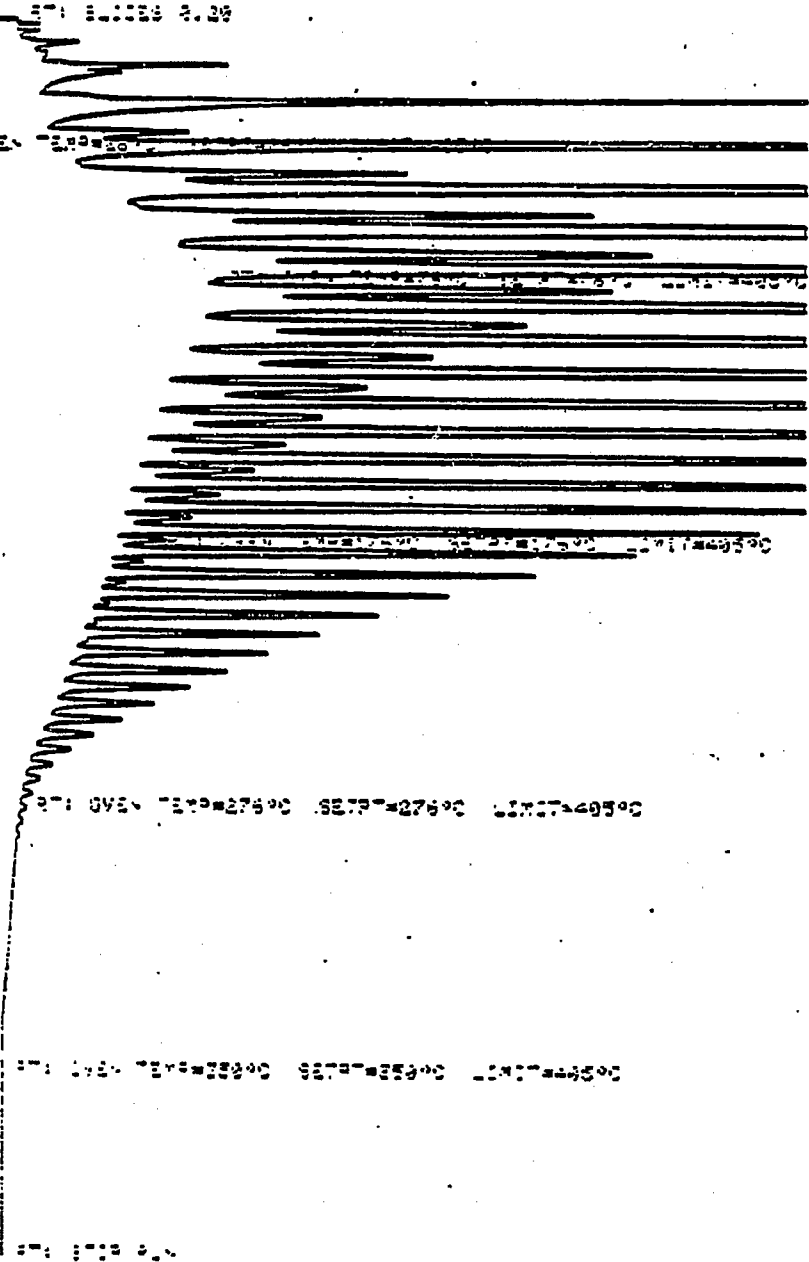
Fig. B89

194

OVER TEMP NOT READY

START: 11:00:00

END: 11:00:00



START: 11:00:00 END: 11:00:00 LIMIT: 405°C

START: 11:00:00 END: 11:00:00 LIMIT: 405°C

END: 11:00:00

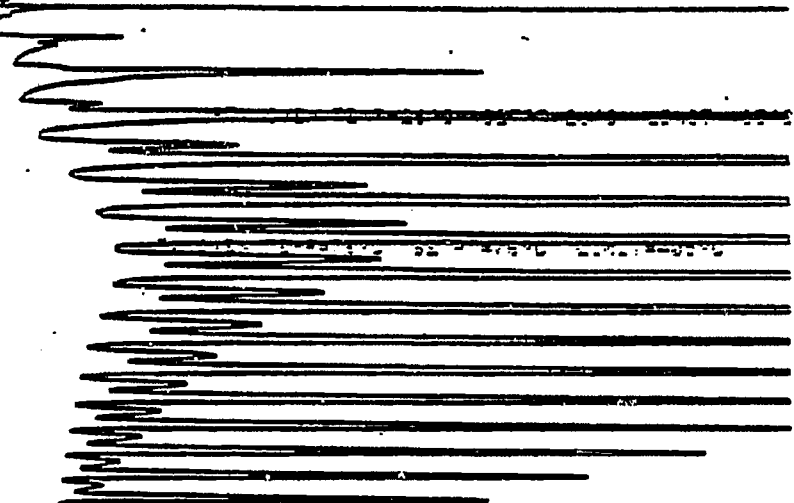
12135-07-07

Fig. B90

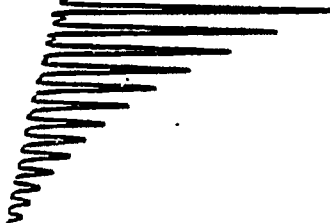
OVEN TEMP NOT READY

CCT

SET POINT 375°C



SET POINT 375°C SET POINT 375°C LIMIT 405°C



OVEN TEMP 375°C SET POINT 375°C LIMIT 405°C

SET POINT 375°C SET POINT 375°C LIMIT 405°C

SET POINT 375°C

12185-07-08

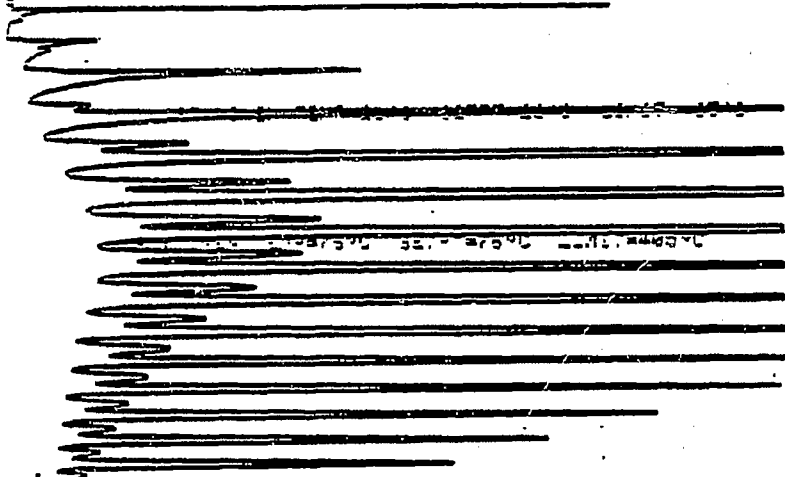
12185-07-08

Fig. B91

000

OVEN TEMP NOT READY

0.20



TEMP=175°C SETPT=175°C LIMIT=405°C

TEMP=275°C SETPT=275°C LIMIT=405°C

TEMP=375°C SETPT=375°C LIMIT=405°C

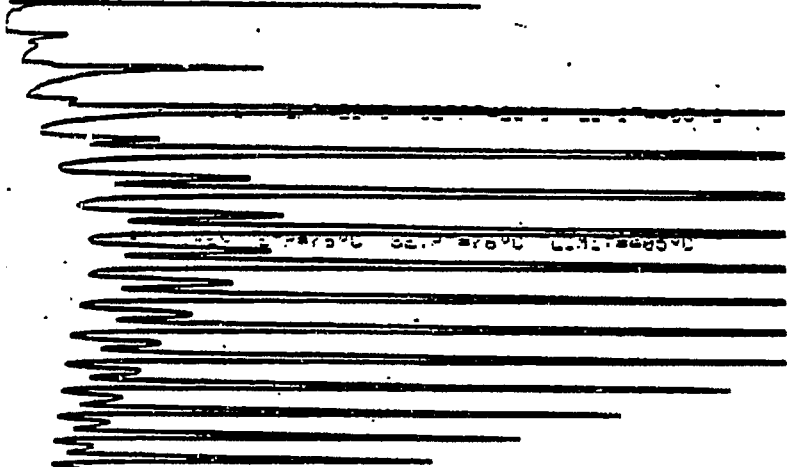
12185-07-09

Fig. B92

OVER TIME NOT READY

ATC

BT: SLICES 0.25



BT: 063 TEMP=175°C SETPT=175°C LIMIT=485°C

BT: 075 TEMP=275°C SETPT=275°C LIMIT=485°C

BT: 075 TEMP=325°C SETPT=325°C LIMIT=485°C

BT: 075 0.25

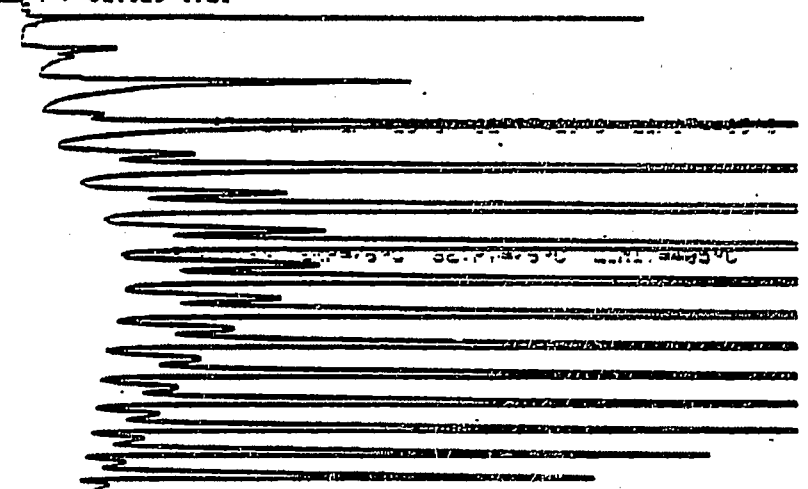
12185-07-10
3000-11:11:25-7-10

Fig. B93

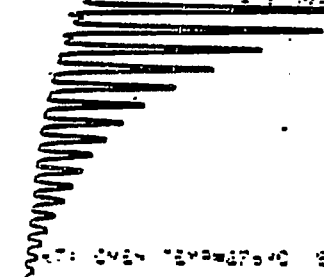
TTU

OVEN TEMP. NOT READY

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BT: 511025 3.29 SETPT=17500 LIMIT=49500



BT: 511025 3.29 SETPT=17500 LIMIT=49500

BT: 511025 3.29 SETPT=17500 LIMIT=49500

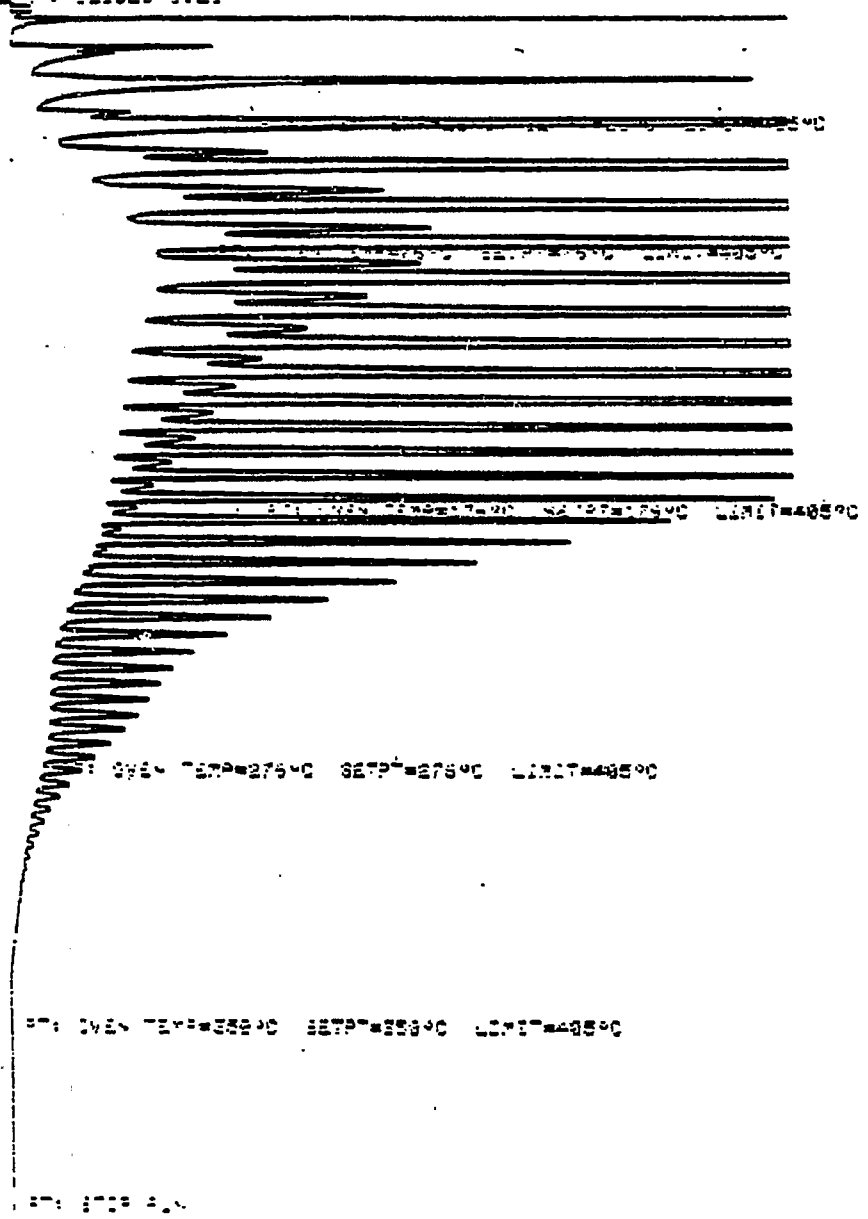
12:95-07-11

Fig. B94

710

OVER TEMP NOT REPLY

ST: 2.1028 2.22



12:05-07-12

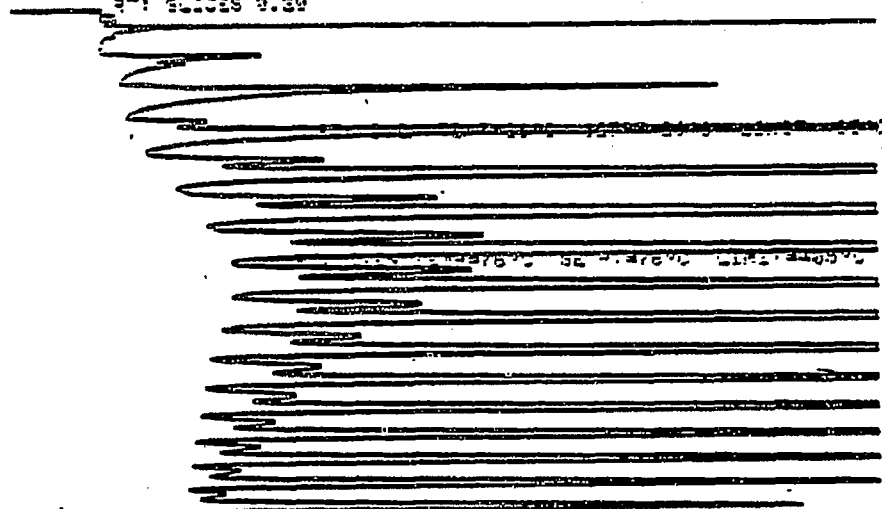
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Fig. B95

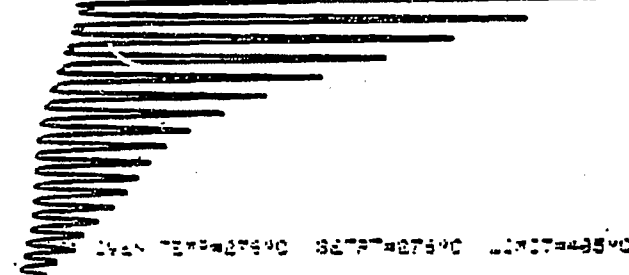
OVEN TEMP NOT READY

CTO

PT: 01005 0.30



PT: OVEN TEMP NOT READY



PT: 01005 0.30

PT: 01005 0.30

PT: 01005 0.30

12195-07-13

Fig. B96

RESULT OF SYNGAS OPERATION

RUN NO. 12185-07
 CATALYST CO/TH/K4-U103+U101 12006-59 250 CC 111.15 G (128.5 G AFTER RUN)
 FEED H2:CO OF 50:50 @ 1260 CC/MIN OR 300 GHSV

RUN & SAMPLE NO.	12185-07-01	185-07-02	185-07-03	185-07-04	185-07-05
FEED H2:CO:AR	50:50: 0	50:50: 0	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	19.0	43.0	67.0	95.5	211.5
PRESSURE, PSIG	300	300	300	300	300
TEMP. C	261	261	260	260	260
FEED CC/MIN	1260	1260	1260	1260	1260
HOURS FEEDING	19.00	24.00	24.00	28.50	116.00
EFFLNT GAS LITER	700.35	957.25	981.45	1193.45	5101.00
GM AQUEOUS LAYER	158.59	189.57	185.49	218.36	925.96
GM OIL	38.86	53.92	55.77	75.81	279.22
MATERIAL BALANCE					
GM ATOM CARBON %	84.74	90.00	90.84	95.63	96.17
GM ATOM HYDROGEN %	91.01	95.01	96.04	101.02	99.62
GM ATOM OXYGEN %	93.44	95.79	96.03	96.81	97.50
RATIO CHX/(H2O+CO2)	0.7408	0.8198	0.8345	0.9622	0.9542
RATIO X IN CHX	2.3935	2.4007	2.3985	2.3702	2.3736
USAGE H2/CO PRDCT	2.2874	2.1899	2.1862	2.0454	2.0744
FEED H2/CO FRM EFFLNT	1.0741	1.0556	1.0572	1.0563	1.0359
RESIDUAL H2/CO RATIO	0.5111	0.5378	0.5563	0.5636	0.5834
RATIO CO2/(H2O+CO2)	0.0584	0.0592	0.0553	0.0566	0.0498
K SHIFT IN EFFLNT	0.0317	0.0338	0.0326	0.0338	0.0306
SPECIFIC ACTIVITY SA	1.1816	1.0833	1.0623	1.1579	0.9857
CONVERSION					
ON CO %	31.69	31.34	30.73	33.25	30.35
ON H2 %	67.50	65.02	63.55	64.38	60.77
ON CO+H2 %	50.24	48.64	47.60	49.24	45.83
PRDCT SELECTIVITY, WT %					
CH4	13.82	14.16	13.94	12.32	12.93
C2 HC'S	2.92	2.92	2.94	2.49	2.66
C3H8	4.06	4.00	4.09	4.38	4.11
C3H6=	2.87	2.83	3.15	3.25	2.98
CAH10	3.21	3.13	3.18	3.75	3.02
CAH8=	5.24	5.20	5.26	5.91	4.50
CSH12	3.82	3.64	3.63	3.87	3.41
CSH10=	4.68	4.52	4.58	5.24	4.63
C6H14	4.09	4.02	3.93	3.82	3.48
C6H12= & CYCLO'S	2.16	2.96	2.83	3.44	3.42
C7+ IN GAS	16.50	14.56	12.93	12.00	16.12
LIQ HC'S	36.62	38.04	39.55	39.54	38.74
TOTAL	100.00	100.00	100.00	100.00	100.00

Table B5

SUB-GROUPING					
C1 -C4	32.13	32.26	32.56	32.08	30.21
C5 -420 F	49.20	46.82	45.81	46.76	49.07
420-700 F	17.03	17.61	17.99	17.87	17.39
700-END PT	1.65	3.31	3.64	3.28	3.33
C5+-END PT	67.87	67.74	67.44	67.92	69.79
ISO/NORMAL MOLE RATIO					
C4	0.1135	0.0874	0.0858	0.1129	0.0558
C5	0.2006	0.1515	0.1411	0.1249	0.1038
C6	0.2966	0.2364	0.1895	0.0759	0.1228
C4+	0.0547	0.0654	0.0633	0.0751	0.0717
PARAFFIN/OLEFIN RATIO					
C3	1.3479	1.3469	1.2416	1.2857	1.3149
C4	0.5913	0.5810	0.5827	0.6121	0.6475
C5	0.7949	0.7839	0.7703	0.7172	0.7155
SCHULZ-FLORY DISTRBTN					
ALPHA (EXP(SLOPE))	0.7724	0.8018	0.8048	0.7970	0.7977
RATIO CH4/(1-A)**2	2.6673	3.6044	3.6590	2.9893	3.1606
ALPHA FRM CORRELATION	0.8434	0.8410	0.8394	0.8387	0.8370
ALPHA (EXPTL/CORR)	0.9158	0.9534	0.9589	0.9502	0.9530
WACHA FRM CORRELATION	16.5927	17.3628	17.6464	17.8438	18.3662
WACHA (EXPTL/CORR)	0.8327	0.8158	0.7898	0.6904	0.7041
LIQ HC COLLECTION					
PHYS. APPEARANCE	CLD OIL	CLR OIL	CLR OIL	CLR OIL	CLR OIL
DENSITY (* 40 C)	0.7548	0.7567	0.7557	0.7440*	0.7432*
N, REFRACTIVE INDEX	1.4259	1.4272	1.4267	1.4214*	1.4205*
SIMULT'D DISTILATN					
10 WT % @ DEG F	262	271	276	279	284
16	300	302	302	301	302
50	423	449	447	441	442
84	596	644	642	623	623
90	641	688	690	683	684
RANGE(16-84 %)	296	342	340	322	321
WT % @ 420 F	49.00	45.00	45.30	46.50	46.50
WT % @ 700 F	95.50	91.30	90.80	91.70	91.40

Table B5, cont

RESULT OF SYNGAS OPERATION

RUN NO. 12185-07
 CATALYST CO/TH/X4-U103+U101 12006-59 250 CC 111.15 G (128.5 G AFTER RUN)
 FEED H2:CO OF 50:50 @ 1260 CC/MN OR 300 GHSV

RUN & SAMPLE NO.	12185-07-06	185-07-07	185-07-08	185-07-09	185-07-10
FEED H2:CO:AR	50:50: 0	50:50: 0	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	235.5	259.5	379.5	403.5	427.5
PRESSURE, PSIG	300	300	300	300	300
TEMP. C	260	260	260	260	260
FEED CC/MIN	1260	1260	1260	1260	1260
HOURS FEEDING	24.00	24.00	120.00	24.00	24.00
EFFLNT GAS LITER	1082.40	1089.00	5622.45	1133.60	1138.00
GM AQUEOUS LAYER	165.59	162.44	780.33	152.22	152.68
GM OIL	53.34	52.51	252.60	46.81	45.95
MATERIAL BALANCE					
GM ATOM CARBON %	96.63	96.39	98.13	94.32	94.56
GM ATOM HYDROGEN %	99.26	99.14	100.60	99.46	99.90
GM ATOM OXYGEN %	98.24	97.81	98.42	96.79	97.04
RATIO CHX/(H2O+CO2)	0.9430	0.9489	0.9893	0.9060	0.9060
RATIO X IN CHX	2.3866	2.3889	2.3900	2.4115	2.4213
USAGR H2/CO PRDCT	2.0887	2.0884	2.0581	2.1503	2.1543
FEED H2/CO FRM EFFLNT	1.0272	1.0286	1.0252	1.0544	1.0565
RESIDUAL H2/CO RATIO	0.5913	0.6002	0.6161	0.6604	0.6612
RATIO CO2/(H2O+CO2)	0.0504	0.0491	0.0478	0.0458	0.0460
K SHIFT IN EFFLNT	0.0314	0.0310	0.0309	0.0317	0.0319
SPECIFIC ACTIVITY SA	0.9204	0.8886	0.8430	0.6975	0.6964
CONVERSION					
ON CO %	29.11	28.79	28.37	26.45	26.47
ON H2 %	59.19	58.45	56.95	53.93	53.98
ON CO+H2 %	44.35	43.83	42.84	40.55	40.60
PRDCT SELECTIVITY, WT %					
CH4	13.58	13.70	13.58	14.84	15.16
C2 HC'S	2.88	2.76	2.81	2.80	3.19
C3H8	4.22	4.26	4.53	4.40	4.51
C3H6=	2.95	2.92	3.47	2.96	2.92
C4H10	3.12	3.15	3.32	3.28	3.41
C4H8=	4.63	4.64	4.71	4.38	4.56
C5H12	3.46	3.53	3.60	3.56	3.66
C5H10=	4.71	4.75	4.03	4.13	4.00
C6H14	3.45	3.56	4.14	3.54	3.53
C6H12= & CYCLO'S	3.41	3.38	3.47	3.48	3.49
C7+ IN GAS	16.46	16.37	17.00	16.06	15.82
LIQ HC'S	37.12	36.98	35.34	36.57	35.75
TOTAL	100.00	100.00	100.00	100.00	100.00

Table B6

SUB-GROUPING					
C1 -CA	31.40	31.44	32.42	32.66	33.75
C5 -420 F	48.93	48.97	48.92	48.32	46.83
420-700 F	16.55	16.46	15.48	15.61	15.98
700-END PT	3.12	3.14	3.18	3.40	3.43
C5+-END PT	68.60	68.56	67.58	67.34	66.25
ISO/NORMAL MOLE RATIO					
CA	0.0515	0.0604	0.0500	0.0509	0.0500
C5	0.1001	0.1060	0.0950	0.0717	0.0695
C6	0.1263	0.1133	0.2551	0.1168	0.1162
CA=	0.0727	0.0718	0.0738	0.0779	0.0811
PARAFFIN/OLEFIN RATIO					
C3	1.3636	1.3952	1.2439	1.4176	1.4716
C4	0.6508	0.6559	0.6812	0.7229	0.7225
C5	0.7146	0.7219	0.8687	0.8368	0.8888
SCHULZ-FLORY DISTRBTN					
ALPHA (EXP(SLOPE))	0.7937	0.7944	0.7899	0.7917	0.7922
RATIO CH4/(1-A)**2	3.1905	3.2394	3.0761	3.4185	3.5099
ALPHA FRM CORRELATION					
ALPHA (EXPTL/CORR)	0.8364	0.8357	0.8344	0.8310	0.8309
ALPHA (EXPTL/CORR)					
ALPHA (EXPTL/CORR)	0.9489	0.9506	0.9467	0.9527	0.9534
W%CH4 FRM CORRELATION					
W%CH4 (EXPTL/CORR)	18.5691	18.7942	19.1893	20.2398	20.2584
W%CH4 (EXPTL/CORR)	0.7316	0.7288	0.7076	0.7331	0.7483
LIQ HC COLLECTION					
PHYS. APPEARANCE					
CLR OIL	CLR OIL	OIL WAX	OIL WAX	OIL WAX	OIL WAX
DENSITY (* 40 C)	0.7432*	0.7430*	0.7429*	0.7430*	0.7430*
N, REFRACTIVE INDEX	1.4204*	1.4202*	1.4201*	1.4194*	1.4191*
SIMULT'D DISTILATN					
10 WT % @ DEG F	288	289	290	289	293
16	302	302	302	301	308
50	441	442	440	436	444
84	620	621	621	623	634
90	682	683	684	689	696
RANGE(16-84 %)	318	319	319	322	326
WT % @ 420 F					
WT % @ 420 F	47.00	47.00	47.20	48.00	45.70
WT % @ 700 F					
WT % @ 700 F	91.60	91.50	91.00	90.70	90.40

Table B6, cont

RESULT OF SYNGAS OPERATION

RUN NO. 12185-07
 CATALYST CO/TH/X4-U103+U101 12006-59 250 CC 111.15 G (128.5 G AFTER RUN)
 FEED H2:CO OF 50:50 @ 1260. CC/MN OR 300 GHSV

RUN & SAMPLE NO.	12185-07-11	185-07-12	185-07-13
	=====	=====	=====
FEED H2:CO:AR	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	452.0	475.5	499.5
PRESSURE, PSIG	500	500	500
TEMP. C	260	260	259
FEED CC/MIN	1260	1260	1260
HOURS FEEDING	24.50	22.50	25.00
EFFLNT GAS LITER	1065.70	988.54	1141.36
GM AQUEOUS LAYER	174.45	159.35	169.85
GM OIL	56.12	54.92	60.80
MATERIAL BALANCE			
GM ATOM CARBON %	93.38	93.20	95.93
GM ATOM HYDROGEN %	97.09	98.60	99.91
GM ATOM OXYGEN %	96.77	96.32	97.79
RATIO CHX/(H2O+CO2)	0.8832	0.8916	0.9335
RATIO X IN CHX	2.3792	2.3831	2.3814
USAGE H2/CO PRDCT	2.1455	2.1465	2.1047
FEED H2/CO FRM EFFLNT	1.0398	1.0579	1.0415
RESIDUAL H2/CO RATIO	0.5881	0.6121	0.6181
RATIO CO2/(H2O+CO2)	0.0495	0.0472	0.0473
K SHIFT IN EFFLNT	0.0306	0.0303	0.0307
SPECIFIC ACTIVITY SA	0.5138	0.4842	0.4934
CONVERSION			
ON CO %	29.00	29.06	28.48
ON H2 %	59.84	58.95	57.55
ON CO+H2 %	44.72	44.43	43.31
PRDCT SELECTIVITY, WT %			
CH4	13.20	13.29	13.22
C2 HC'S	2.52	2.71	2.46
C3H8	4.18	4.09	4.21
C3H6=	3.28	3.16	3.23
C4H10	3.25	3.16	3.25
C4H8=	4.82	3.65	3.70
C5H12	3.29	3.20	3.25
C5H10=	4.09	3.90	3.92
C6H14	3.35	3.22	3.32
C6H12= & CYCLO'S	3.54	3.38	3.42
C7+ IN GAS	14.63	13.92	14.30
LIQ HC'S	39.85	42.33	41.72
TOTAL	100.00	100.00	100.00

Table B7

SUB-GROUPING			
C1 -C4	31.24	30.06	30.06
C5 -420 F	47.04	46.24	46.20
420-700 F	18.13	18.88	18.32
700-END PT	3.59	4.83	5.42
C5+-END PT	68.76	69.94	69.94
ISO/NORMAL MOLE RATIO			
C4	0.0563	0.0569	0.0543
C5	0.0913	0.0878	0.0874
C6	0.1148	0.0999	0.1045
C4=	0.0634	0.0800	0.0774
PARAFFIN/OLEFIN RATIO			
C3	1.2179	1.2346	1.2432
C4	0.6508	0.8346	0.8490
C5	0.7819	0.7974	0.8048
SCHULZ-FLORY DISTRBTM			
ALPHA (EXP(SLOPE))	0.7931	0.8064	0.8085
RATIO CH4/(1-A)**2	3.0832	3.5446	3.6048
ALPHA FRM CORRELATION	0.8367	0.8347	0.8343
ALPHA (EXPTL/CORR)	0.9480	0.9661	0.9691
W%CH4 FRM CORRELATION	18.4878	19.0915	19.0114
W%CH4 (EXPTL/CORR)	0.7137	0.6961	0.6951
LIQ HC COLLECTION			
PHYS. APPEARANCE	OIL WAX	OIL WAX	OIL WAX
DENSITY (* 40 C)	0.7440*	0.7620	0.760
N, REFRACTIVE INDEX	1.4198*	1.4204*	1.4210*
SIMULT'D DISTILATM			
10 WT % @ DEG F	293	291	293
16	303	302	305
50	445	449	451
84	621	658	666
90	682	720	736
RANGE(16-84 %)	318	356	361
WT % @ 420 F	45.50	44.00	43.10
WT % @ 700 F	91.00	88.60	87.00
	45.50	44.00	43.11
	91.09	88.56	87.00

Table B7, cont

V. Run 13 (12200-07) with Catalyst 13 (Co/Th/X₄/UCC-103)

The purpose of this run was to try to isolate the effect of UCC-101 in Catalyst 12. As in the preparation of Catalysts 10 and 12, the thorium-promoted cobalt oxide was formed in close contact with UCC-103, then further promoted with X₄. The resulting powder, after bonding with 15 percent silica, was extruded to 1/8-inch pellets. The final catalyst contained 8.3 percent cobalt, 1.1 percent thorium and 0.8 percent X₄.

Conversion, product selectivity, isomerization of the pentane, and percent olefins of the C₄'s are plotted against time on stream in Figs. B97-100. Simulated distillations of the C₅⁺ product are plotted in Figs. B101-110. Carbon number product distributions are plotted in Figs. B111-120. Chromatograms from simulated distillations are reproduced in Figs. B121-130. Detailed material balances appear in Tables B8-9.

The specific activity of this catalyst was about 2.2, or about 1.8 times that of Catalyst 12185-07. But since it also contained about 1.8 times the concentration of cobalt, the activity per gram cobalt was essentially the same.

The stability, however, was considerably poorer. As estimated by linear least squares analysis, the syngas conversion decreased at a rate of one percentage point every 21.8 hours, some three times as rapidly as with Catalyst 12185-07. This may,

however, have been due not so much to the absence of UCC-101 as to a lower residual $H_2:CO$ ratio in the reactor, 0.40 versus 0.53 $H_2:CO$ for Catalyst 12185-07.

The product balance was biased toward the heavier species, which is also consistent with a lower residual $H_2:CO$ ratio. With both catalysts the ratios of experimentally observed methane to methane predicted by the mathematical model are essentially the same at about 0.7:1.

The Schulz-Flory plots of the product distributions are fairly linear except for the usual excess of methane. This is true for both formulations, with and without UCC-101. Isomerization of the pentane was about 6 percent of total pentane produced; with Catalyst 12185-07 it was initially 12 percent and decreased with time on stream.

This run yielded some useful information on the function of UCC-101 as an additive. Its presence apparently has little effect on a catalyst's product distribution. The catalyst lacking UCC-101 was less stable, but this may have been an effect of its higher activity.

RUN 12200-07

518¹CO
300 PSIG
280°C

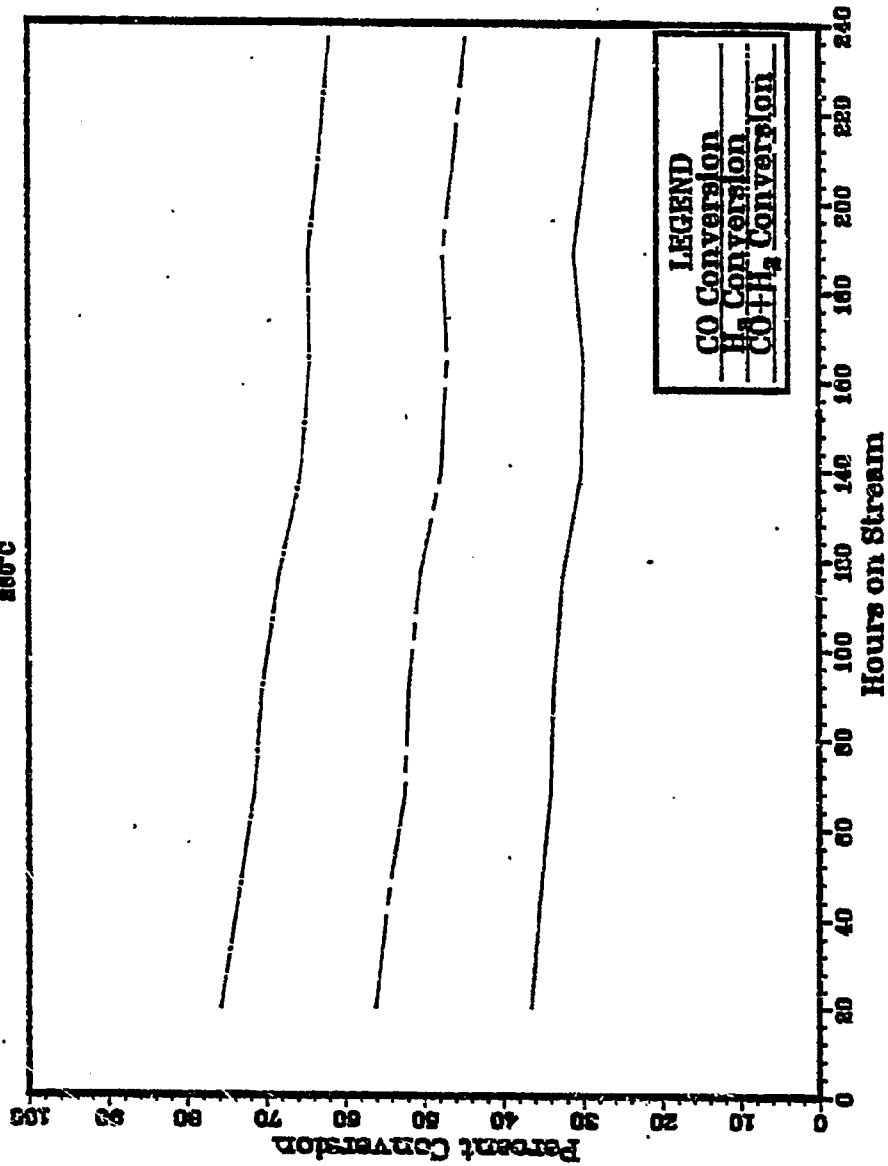


Fig. B97

RUN 12200-07

111E-100
500 PSIG
860°C

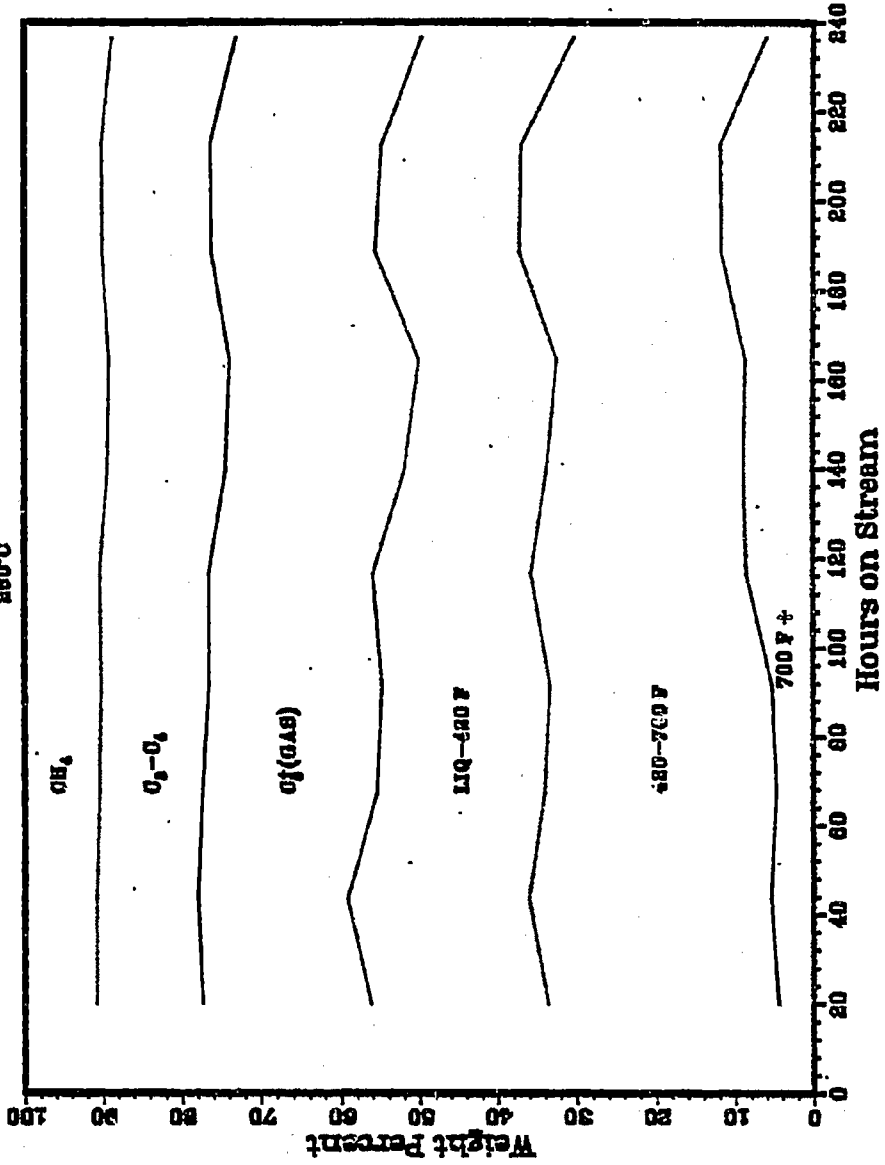


Fig. B98

RUN 12200-07

111 H₂CO
300 PSIG
165°C

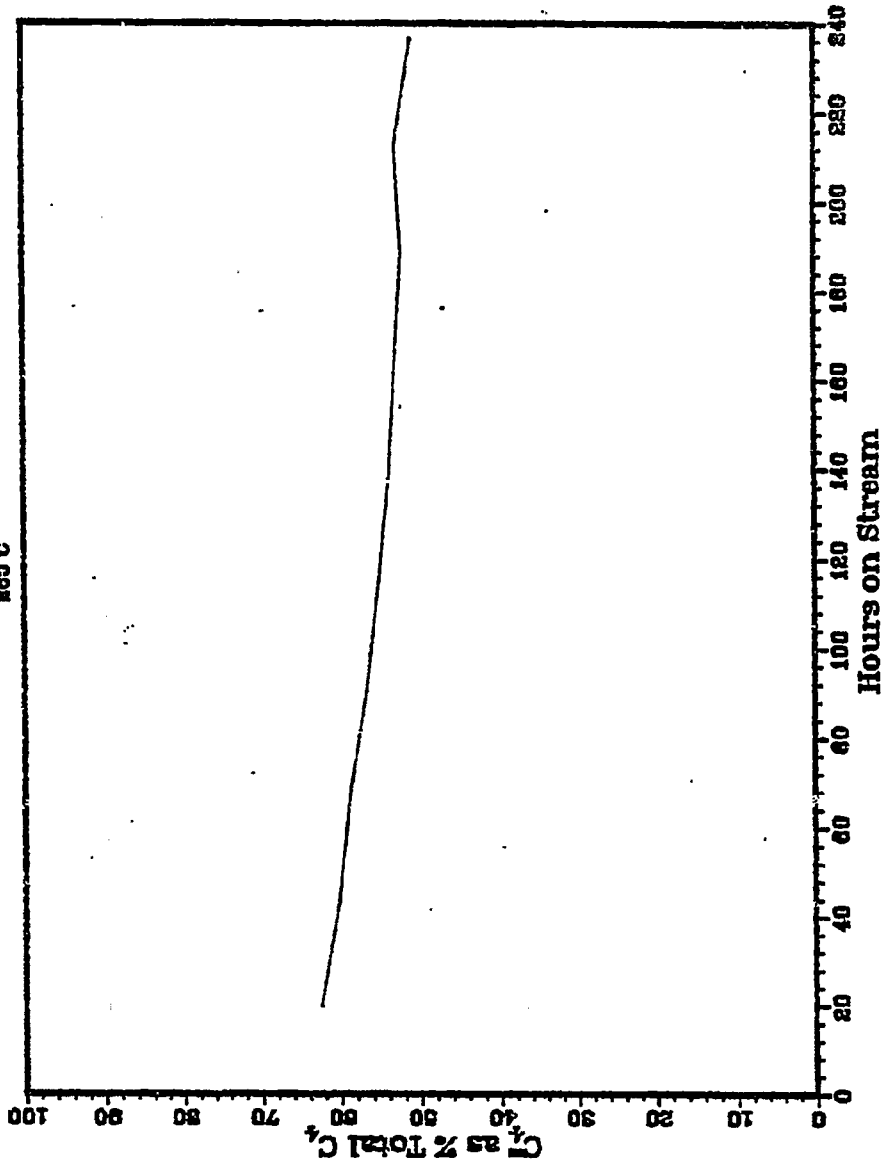


Fig. B99

RUN 12200-07

111 H₂CO
300 FOLD
880°C

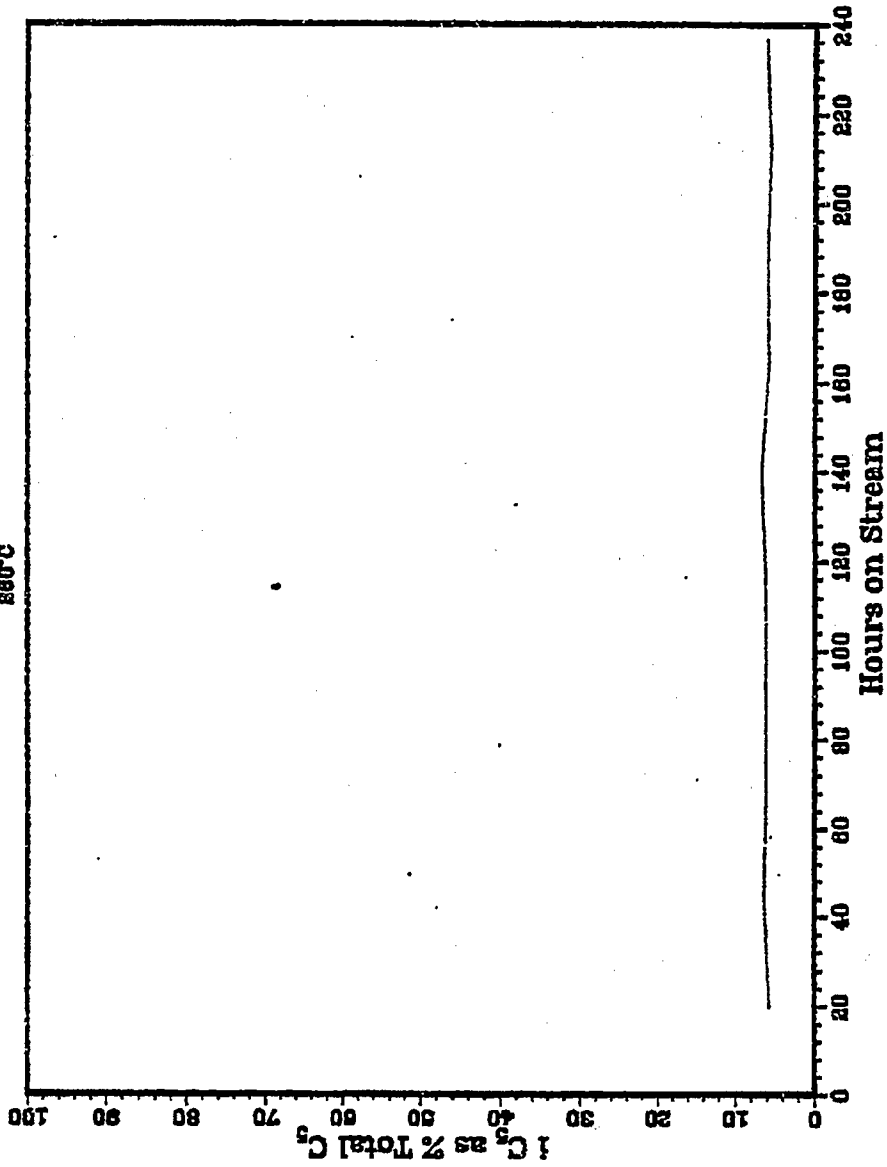


Fig. B100