

Fig. A53

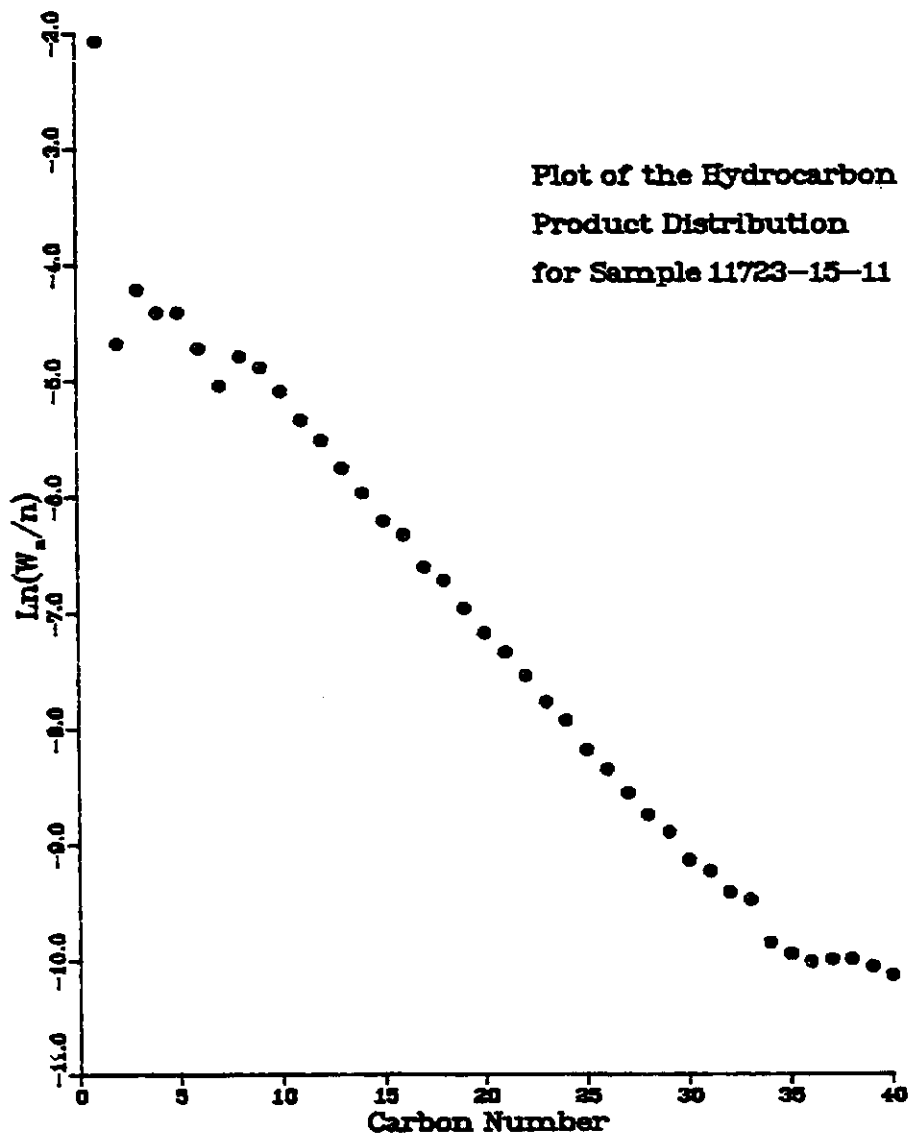


Fig. A54

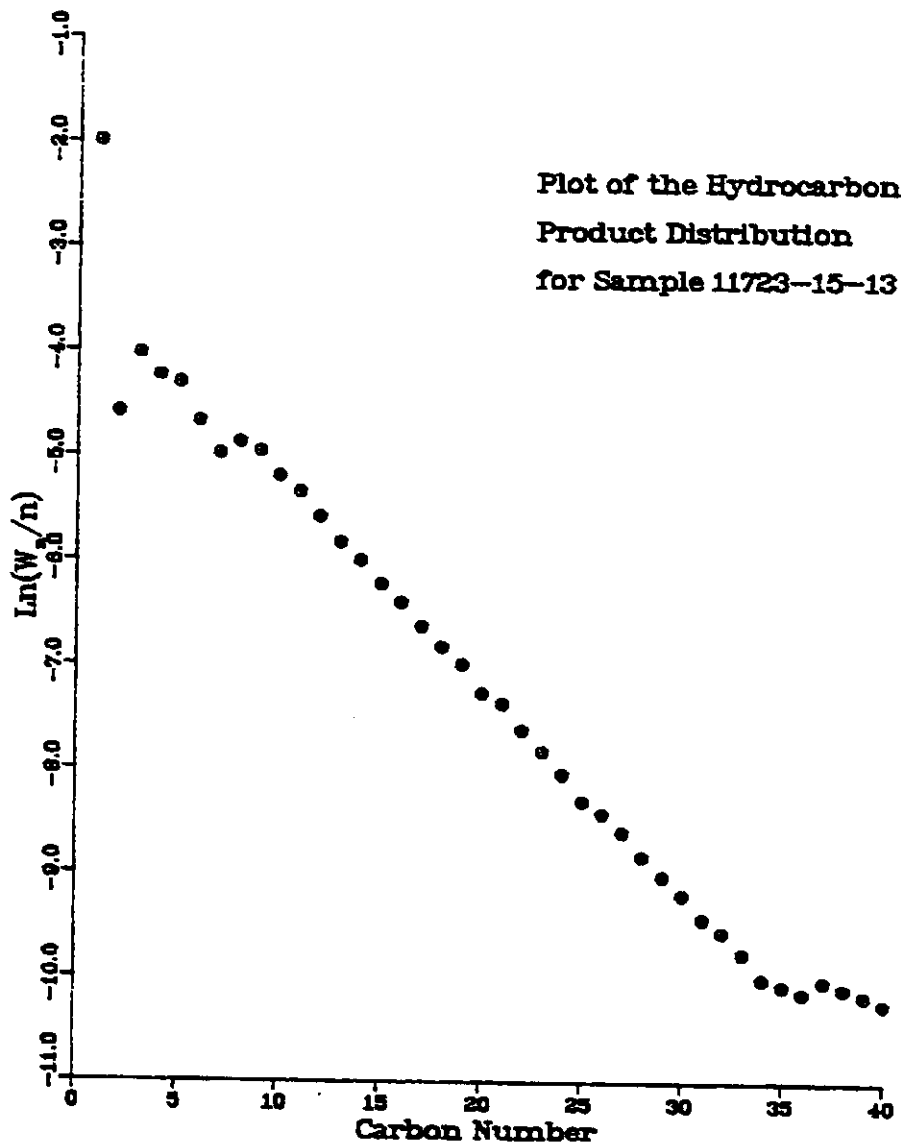


Fig. A55

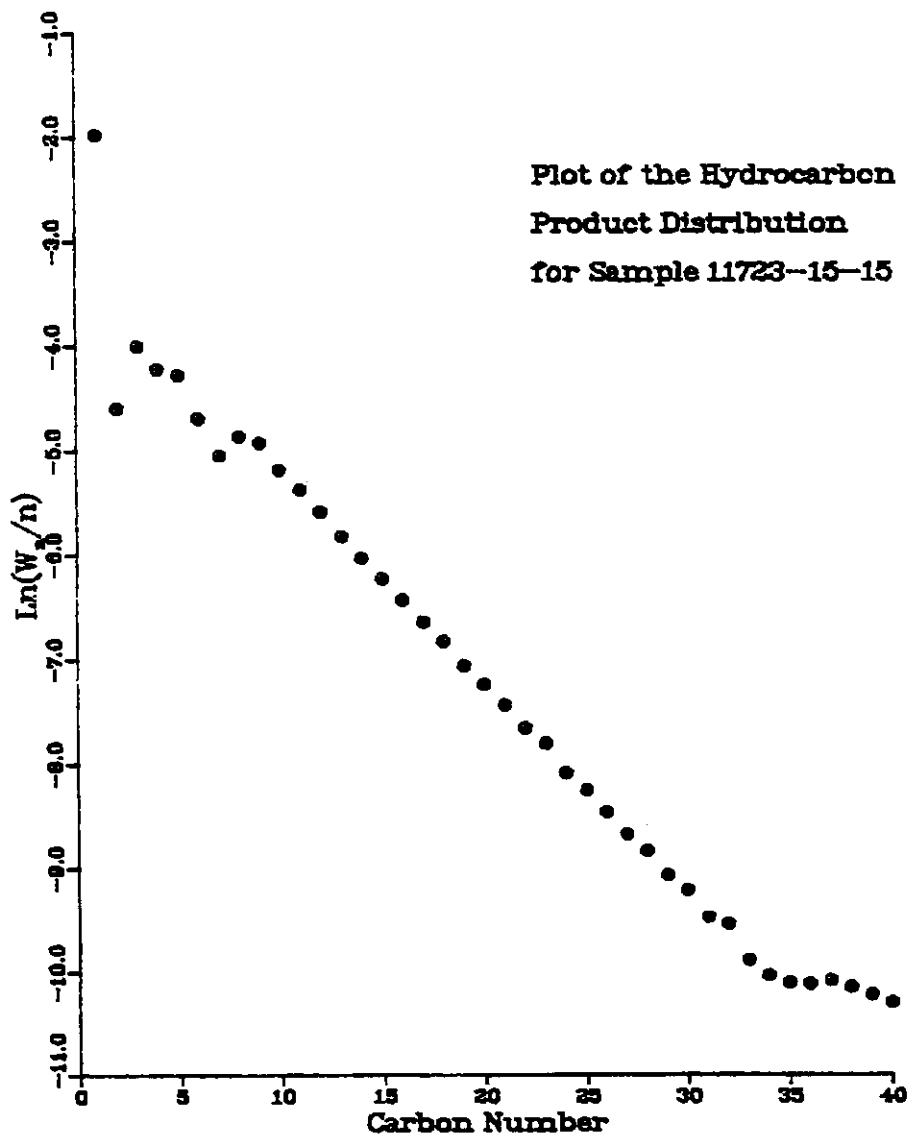


Fig. A56

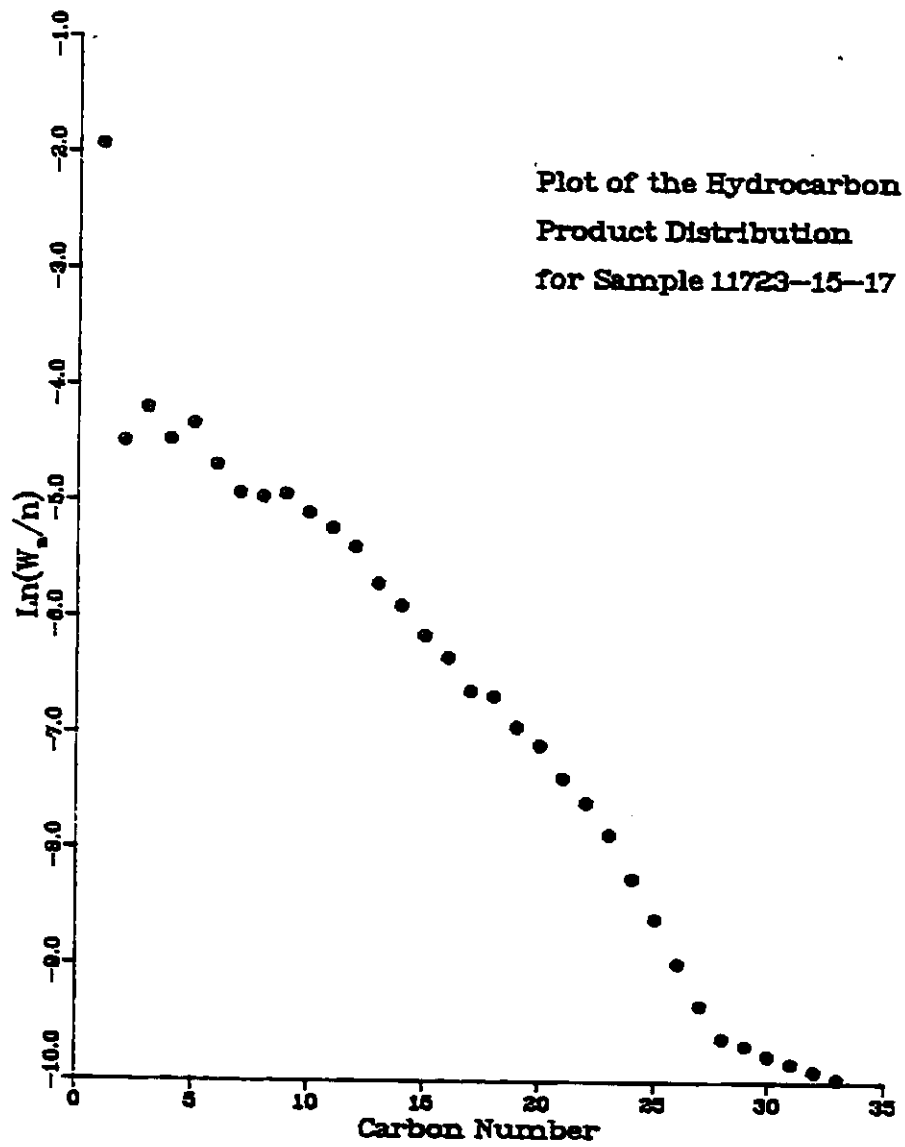


Fig. A57

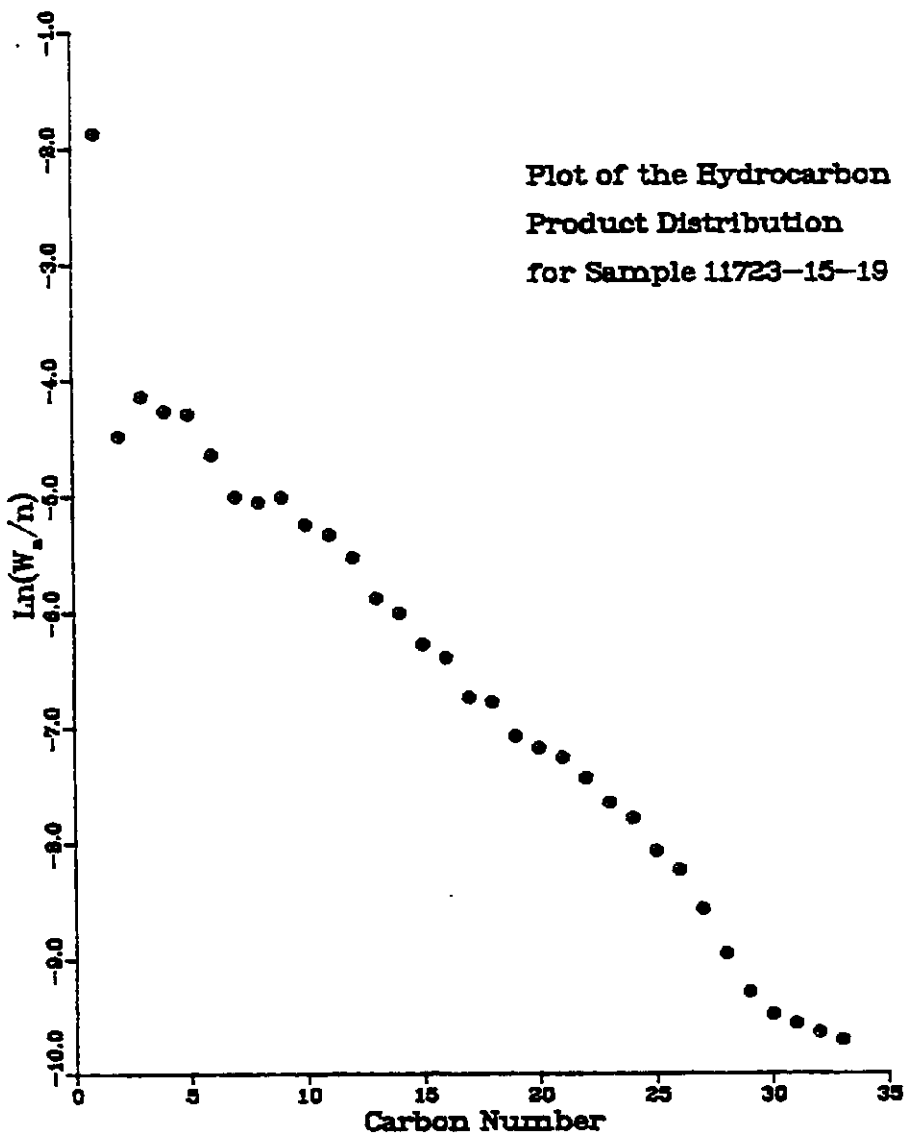


Fig. A58

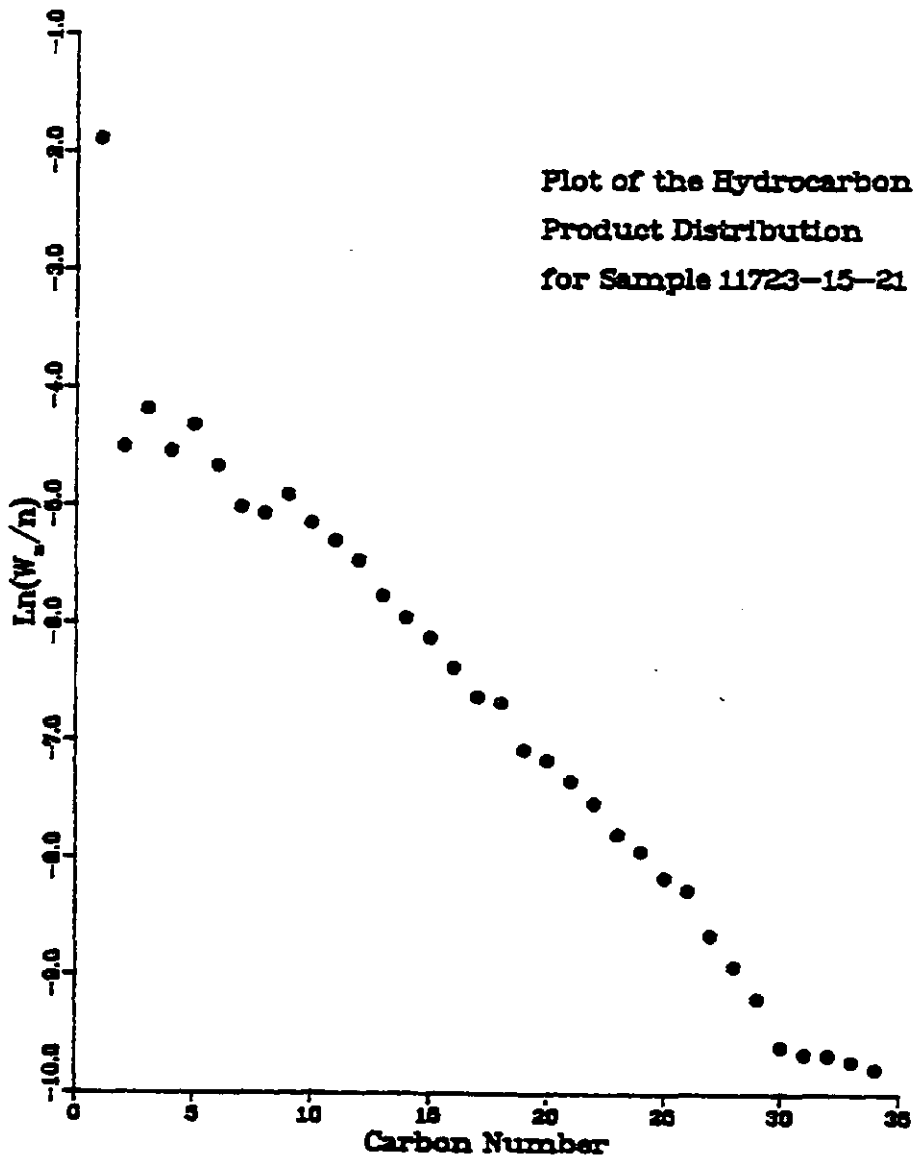


Fig. A59

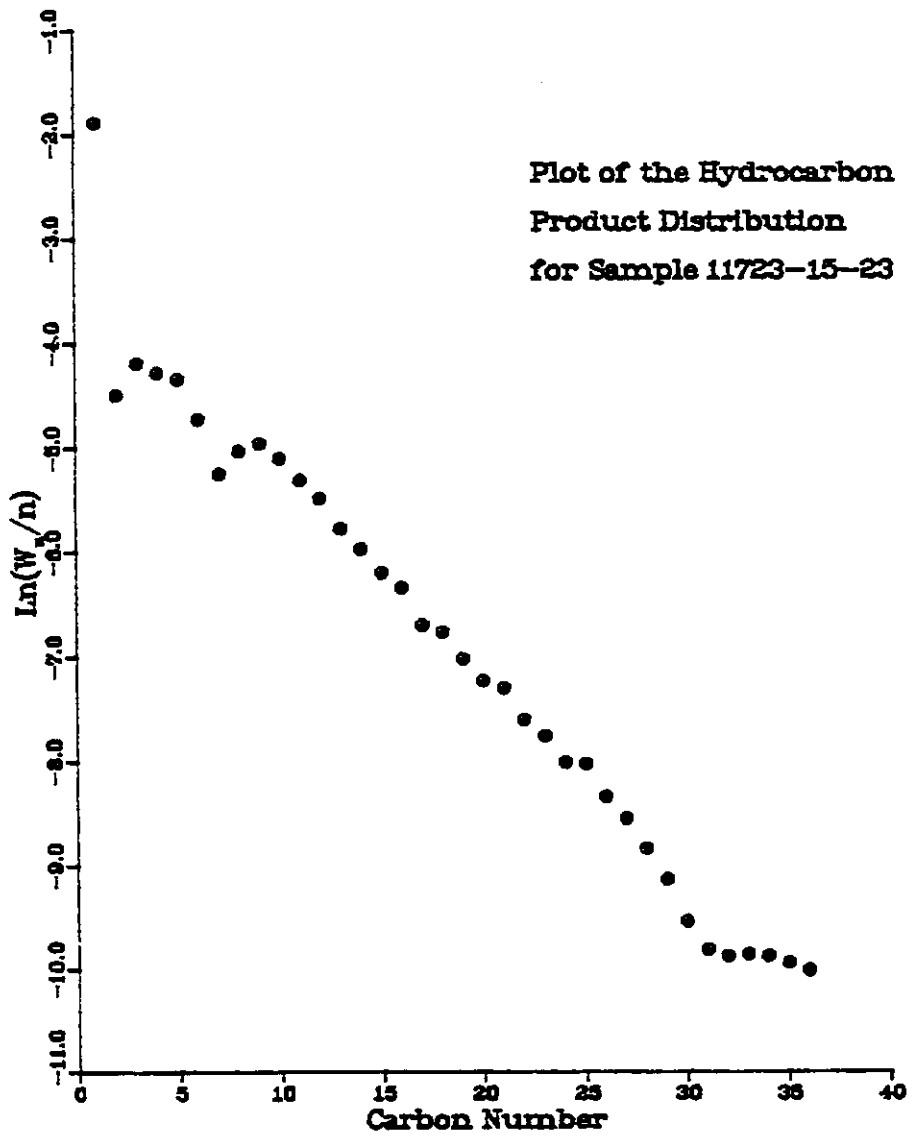
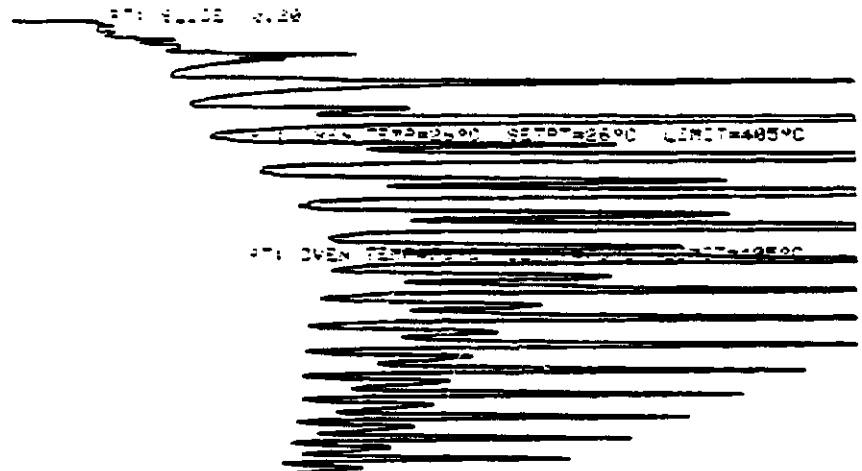


Fig. A60



OVEN TEMP NOT RECD-

RT: 9.115 9.20



OVEN TEMP=26.00 SETPT=26.00 LIMIT=405.00

RT: OVEN TEMP=26.00 SETPT=26.00 LIMIT=405.00

RT: OVEN TEMP=27.00 SETPT=27.00 LIMIT=405.00

RT: OVEN TEMP=27.60 SETPT=27.60 LIMIT=405.00

RT: OVEN TEMP=35.00 SETPT=35.00 LIMIT=405.00

RT: STOP RUN

99091311725-15-4L

Fig. A61

OVEN TEMP NOT READY

RT: ELIDES 6.30

OVEN TEMP=26°C SETPT=26°C LIMIT=485°C

RT: OVEN TEMP=76°C SETPT=76°C LIMIT=485°C

OVEN TEMP=176°C SETPT=176°C LIMIT=485°C

RT: OVEN TEMP=276°C SETPT=276°C LIMIT=485°C

RT: OVEN TEMP=350°C SETPT=350°C LIMIT=485°C

RT: STOP RUN

SAMPLE: 911723-15-7L

Fig. A62

017

OVEN TEMP NOT READY

RT: SUCCESS 2.29

OVEN TEMP=25°C SETPT=25°C LIMIT=405°C

RT: OVEN TEMP=176°C SETPT=176°C LIMIT=405°C

OVEN TEMP=176°C SETPT=176°C LIMIT=405°C

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

RT: OVEN TEMP=350°C SETPT=350°C LIMIT=405°C

RT: STOP RUN

SAMPLE: D11723-15-13

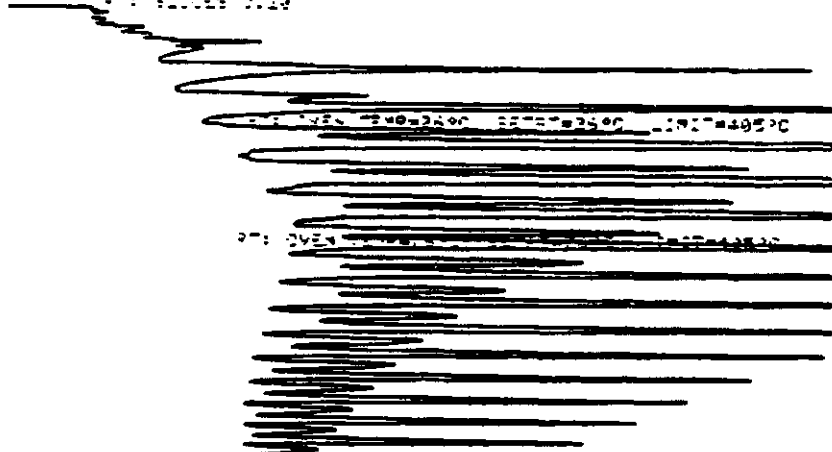
Fig. A63

021

021

OVEN TEMP NOT REACH

RT: 0000: 0000



RT: OVEN TEMP=225°C SETPT=225°C LIMIT=405°C

RT: OVEN TEMP=175°C SETPT=175°C LIMIT=405°C

RT: OVEN TEMP=275°C SETPT=275°C LIMIT=405°C

RT: OVEN TEMP=150°C SETPT=150°C LIMIT=405°C

RT: STOP RUN

SAMPLE: 011723-15-19

Fig. A64

03

031

Table A6

## RESULT OF SYNGAS OPERATION

RUN NO.	11723-15				
CATALYST	Co/Th/X8-U103+U101 11864-15C 250 CC 103.6G(142.4 @END +39. G)				
FEED	H2:CO:ARGON OF 50:50:0 @ 1260 CC/MN OR 302 GHSV				
RUN & SAMPLE NO.	11723-15-02	723-15-04	723-15-05	723-15-07	723-15-09
FEED H2:CO:AR	50:50: 0	50:50: 0	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	23.3	47.3	71.3	95.3	119.0
PRESSURE, PSIG	298	300	302	304	301
TEMP. C	266	264	269	267	262
FEED CC/MIN	1260	1260	1260	1260	1260
HOURS FEEDING	23.00	24.00	16.50	24.00	23.67
EFFLNT GAS LITER	537.08	641.25	483.30	739.55	747.50
GM AQUEOUS LAYER	180.57	213.18	152.14	226.88	227.82
GM OIL	83.71	91.52	81.76	107.92	113.86
MATERIAL BALANCE					
GM ATOM CARBON %	74.91	82.56	94.73	93.12	95.10
GM ATOM HYDROGEN %	78.58	87.64	101.82	98.77	98.89
GM ATOM OXYGEN %	79.43	87.07	94.72	96.46	96.04
RATIO CHX/(H2O+CO2)	0.8776	0.8843	1.0003	0.9205	0.9269
RATIO X IN CHX	2.3250	2.3309	2.3905	2.3880	2.3421
USAGE H2/CO PRDCT	1.6678	1.8097	1.6371	1.7902	1.9122
FEED H2/CO FRM EFFLNT	1.0490	1.0616	1.0749	1.0607	1.0622
RESIDUAL H2/CO RATIO	0.3374	0.3420	0.3656	0.3622	0.3740
RATIO CO2/(H2O+CO2)	0.2086	0.1532	0.2116	0.1617	0.1075
K SHIFT IN EFFLNT	0.0890	0.0618	0.0981	0.0699	0.0450
SPECIFIC ACTIVITY SA	3.8949	3.5891	3.0876	2.7540	2.9986
CONVERSION					
ON CO %	53.49	49.03	55.78	48.92	44.74
ON H2 %	85.04	83.58	84.96	82.56	80.54
ON CO+H2 %	69.64	66.82	70.90	66.23	63.18
PRDT SELECTIVITY, WT %					
CH4	11.69	12.24	14.86	14.67	12.25
C2 HC'S	1.85	1.73	2.55	2.17	1.88
C3H8	2.20	2.00	2.49	2.42	2.06
C3H6=	2.51	2.50	2.19	2.39	2.37
C4H10	1.62	1.56	1.83	1.86	1.55
C4H8=	3.76	3.59	3.61	3.74	3.17
C5H12	1.67	1.60	1.82	1.77	1.60
C5H10=	4.92	4.68	4.37	2.70	4.21
C6H14	2.13	1.92	2.07	2.04	1.73
C6H12= & CYCLO'S	3.93	3.64	3.44	3.63	3.35
C7+ IN GAS	13.38	15.07	10.18	10.85	8.17
LIQ HC'S	50.36	49.48	50.65	51.75	57.66
TOTAL	100.00	100.00	100.00	100.00	100.00

Table A6 (continued)

SUB-GROUPING					
C1 -C4	23.63	23.61	27.48	27.25	23.29
C5 -420 F	51.95	50.16	45.24	44.87	43.70
420-700 F	21.87	22.98	19.79	21.94	24.31
700-END PT	2.55	3.24	7.50	5.93	8.69
C5+-END PT	76.37	76.39	72.52	72.75	76.71
ISO/NORMAL MOLE RATIO					
C4	0.1048	0.0665	0.0870	0.0649	0.0536
C5	0.1245	0.0854	0.1179	0.0900	0.0737
C6	0.4496	0.3260	0.3738	0.2780	0.2041
C4=	0.0617	0.0604	0.0738	0.0693	0.0600
PARAFFIN/OLEFIN RATIO					
C3	0.8340	0.7609	1.0852	0.9666	0.8276
C4	0.4162	0.4193	0.4955	0.4814	0.4709
C5	0.3293	0.3323	0.4037	0.6374	0.3699
SCHULZ-FLORY DISTRBTN					
ALPHA (EXP(SLOPE))	0.7956	0.8125	0.8313	0.8252	0.8405
RATIO CH4/(1-A)**2	2.7967	3.4813	5.2238	4.8005	4.8156
ALPHA FRM CORRELATION					
ALPHA (EXPTL/CORR)	0.8634	0.8629	0.8593	0.8599	0.8586
	0.9215	0.9416	0.9674	0.9596	0.9789
W%CH4 FRM CORRELATION					
W%CH4 (EXPTL/CORR)	11.4404	11.1947	13.3156	12.7321	12.0987
	1.0214	1.0935	1.1160	1.1524	1.0125
LIQ HC COLLECTION					
PHYS. APPEARANCE	CLDY&SLD	CLDY	YLW WAXY	YLW WAXY	YLW WAXY
DENSITY	0.756	0.758	0.762	0.756	0.763
N, REFRACTIVE INDEX	1.4272	1.4275	1.4310	1.4295	1.4305
SIMULT'D DISTILAIN					
10 WT % @ DEG F	253	256	256	259	265
16	283	294	294	298	301
50	415	435	444	443	456
84	595	623	687	653	688
90	644	668	759	719	760
RANGE(16-84 %)	312	329	393	355	387
WT % @ 420 F	51.50	47.00	46.13	46.14	42.75
WT % @ 700 F	94.93	93.45	85.20	88.54	84.92

REMARKS:

HIGH TEMP

NEW FORMAT JAN 25,85

Table A7

## RESULT OF SYNGAS OPERATION

RUN NO. 11723-15  
 CATALYST Co/Th/X8-U103+U101 11864-15C 250 CC 103.6G(142.4 @END +39. G)  
 FEED H<sub>2</sub>:CO:ARGON OF 50:50:0 @ 1260 CC/MN OR 302 GHSV

RUN & SAMPLE NO.	11723-15-11	723-15-13	723-15-15	723-15-17	723-15-19
FEED H <sub>2</sub> :CO:AR	50:50: 0	50:50: 0	50:50: 0	50:50: 0	50:50: 0
HRS ON STREAM	143.5	166.3	197.5	221.5	245.5
PRESSURE, PSIG	300	300	302	298	303
TEMP. C	264	264	264	264	263
FEED CC/MIN	1260	1260	1260	1260	1260
HOURS FEEDING	24.50	22.83	25.00	24.00	24.00
EFFLNT GAS LITER	784.53	756.38	839.19	812.55	815.75
GM AQUEOUS LAYER	230.82	213.87	232.67	221.88	220.90
GM OIL	113.40	102.46	108.85	105.49	101.80
MATERIAL BALANCE					
GM ATOM CARBON %	92.43	96.01	95.92	95.24	95.89
GM ATOM HYDROGEN %	98.87	101.25	101.06	100.26	101.25
GM ATOM OXYGEN %	94.87	97.25	97.36	97.49	97.83
RATIO CH <sub>x</sub> /(H <sub>2</sub> O+CO <sub>2</sub> )	0.9378	0.9689	0.9636	0.9423	0.9510
RATIO X IN CH <sub>x</sub>	2.3484	2.3683	2.3726	2.3862	2.4032
USAGE H <sub>2</sub> /CO PRDCT	1.9220	1.8648	1.8809	1.9083	1.8703
FEED H <sub>2</sub> /CO FRM EFFLNT	1.0697	1.0545	1.0536	1.0527	1.0559
RESIDUAL H <sub>2</sub> /CO RATIO	0.3957	0.3884	0.3950	0.4003	0.4033
RATIO CO <sub>2</sub> /(H <sub>2</sub> O+CO <sub>2</sub> )	0.1022	0.1188	0.1148	0.1121	0.1268
K SHIFT IN EFFLNT	0.0451	0.0524	0.0512	0.0506	0.0586
SPECIFIC ACTIVITY SA	2.4419	2.6049	2.4579	2.3637	2.5007
CONVERSION					
ON CO %	44.16	45.12	44.32	43.26	44.48
ON H <sub>2</sub> %	79.34	79.79	79.13	78.42	78.79
ON CO+H <sub>2</sub> %	62.34	62.91	62.18	61.29	62.10
PRDCT SELECTIVITY, WT %					
CH <sub>4</sub>	12.62	13.59	13.81	14.46	15.43
C <sub>2</sub> HC'S	1.36	2.03	2.03	2.25	2.26
C <sub>3</sub> H <sub>8</sub>	2.08	2.47	2.47	2.29	2.46
C <sub>3</sub> H <sub>6</sub> =	2.34	2.85	3.01	2.23	2.31
C <sub>4</sub> H <sub>10</sub>	1.58	1.87	2.01	1.75	1.94
C <sub>4</sub> H <sub>8</sub> =	3.27	3.89	3.86	2.81	3.67
C <sub>5</sub> H <sub>12</sub>	1.76	2.10	2.18	1.97	2.12
C <sub>5</sub> H <sub>10</sub> =	4.28	4.62	4.74	4.60	4.73
C <sub>6</sub> H <sub>14</sub>	1.89	2.01	2.04	2.04	2.12
C <sub>6</sub> H <sub>12</sub> = & CYCLO'S	3.44	3.60	3.46	3.44	3.67
C <sub>7</sub> + IN GAS	8.62	9.03	9.23	8.93	9.10
LIQ HC'S	56.24	51.93	51.16	53.24	50.20
TOTAL	100.00	100.00	100.00	100.00	100.00

Table A7 (continued)

SUB-GROUPING					
C1 -C4	23.76	26.71	27.19	25.79	28.06
C5 -420 F	45.50	44.73	44.93	46.17	43.58
420-700 F	24.22	22.69	21.91	24.80	23.04
700-END PT	6.52	5.87	5.97	3.24	5.32
C5+-END PT	76.24	73.29	72.81	74.21	71.94
ISO/NORMAL MOLE RATIO					
C4	0.0522	0.1095	0.0439	0.0453	0.0492
C5	0.0681	0.0825	0.0816	0.0594	0.0870
C6	0.1975	0.2282	0.2181	0.1986	0.2000
C4=	0.0602	0.0781	0.0805	0.0875	0.0687
PARAFFIN/OLEFIN RATIO					
C3	0.8475	0.8276	0.7822	0.9816	1.0159
C4	0.4665	0.4651	0.5015	0.6025	0.5102
C5	0.4006	0.4420	0.4461	0.4153	0.4362
SCHULZ-FLORY DISTRBIN					
ALPHA (EXP(SLOPE))	0.8293	0.8232	0.8216	0.8095	0.8293
RATIO CH4/(1-A)**2	4.3312	4.3507	4.3575	3.9877	5.2966
ALPHA FRM CORRELATION	0.8557	0.8566	0.8558	0.8552	0.8549
ALPHA (EXPTL/CORR)	0.9691	0.9610	0.9600	0.9466	0.9701
W%CH4 FRM CORRELATION	13.4017	13.1191	13.3735	13.5770	13.4640
W%CH4 (EXPTL/CORR)	0.9418	1.0361	1.0327	1.0654	1.1458
LIQ HC COLLECTION					
PHYS. APPEARANCE	GRN WAXY	GRN WAXY	GRN WAXY	GRN WAXY	GRN WAXY
DENSITY	0.753	0.758	0.758	0.754	0.757
N, REFRACTIVE INDEX	1.4290	1.4290	1.4285	1.4274	1.4285
SIMULT'D DISTILATN					
10 WT % @ DEG F	261	262	264	261	265
16	299	300	300	299	303
50	447	448	447	429	451
84	657	653	650	614	655
90	721	715	712	661	706
RANGE(16-84 %)	358	553	350	315	352
WT % @ 420 F	45.33	45.00	45.50	47.33	43.50
WT % @ 700 F	88.40	88.69	88.33	93.91	89.40

NEW FORMAT JAN 25,85



Table A8

## RESULT OF SYNGAS OPERATION

RUN NO. 11723-15  
 CATALYST Co/Th/X8-U103+U101 11864-15C 250 CC 103.6G(142.4 @END +39. G)  
 FEED H<sub>2</sub>:CO:ARGON OF 50:50:0 @ 1260 CC/MN OR 302 GHSV

RUN & SAMPLE NO.	11723-15-21	723-15-23
FEED H <sub>2</sub> :CO:AR	50:50:0	50:50:0
HRS ON STREAM	269.5	293.5
PRESSURE, PSIG	300	300
TEMP. C	262	263
FEED CC/MIN	1260	1260
HOURS FEEDING	24.00	24.00
EFFLNT GAS LITER	833.25	839.95
GM AQUEOUS LAYER	219.43	216.85
GM OIL	98.56	97.87
MATERIAL BALANCE		
GM ATOM CARBON %	94.46	95.22
GM ATOM HYDROGEN %	99.29	99.53
GM ATOM OXYGEN %	97.75	97.77
RATIO CH <sub>x</sub> /(H <sub>2</sub> O+CO <sub>2</sub> )	0.9139	0.9329
RATIO X IN CH <sub>x</sub>	2.3975	2.3999
USAGE H <sub>2</sub> /CO PRODT	1.9632	1.9478
FEED H <sub>2</sub> /CO FRM EFFLNT	1.0511	1.0453
RESIDUAL H <sub>2</sub> /CO RATIO	0.4136	0.4128
RATIO CO <sub>2</sub> /(H <sub>2</sub> O+CO <sub>2</sub> )	0.1017	0.1026
K SHIFT IN EFFLNT	0.0468	0.0472
SPECIFIC ACTIVITY SA	2.2927	2.1939
CONVERSION		
ON CO %	41.14	41.20
ON H <sub>2</sub> %	76.84	76.78
ON CO+H <sub>2</sub> %	59.44	59.58
PRDT SELECTIVITY, WT %		
CH <sub>4</sub>	15.09	15.22
C <sub>2</sub> HC'S	2.20	2.24
C <sub>3</sub> H <sub>8</sub>	2.37	2.38
C <sub>3</sub> H <sub>6</sub> =	2.19	2.18
C <sub>4</sub> H <sub>10</sub>	1.79	1.92
C <sub>4</sub> H <sub>8</sub> =	2.46	3.63
C <sub>5</sub> H <sub>12</sub>	2.12	2.20
C <sub>5</sub> H <sub>10</sub> =	4.49	4.34
C <sub>6</sub> H <sub>14</sub>	2.07	2.02
C <sub>6</sub> H <sub>12</sub> = & CYCLO'S	3.55	3.32
C <sub>7</sub> + IN GAS	9.34	9.15
LIQ HC'S	52.33	51.40
TOTAL	100.00	100.00

Table A8 (continued)

SUB-GROUPING		
C1 -C4	26.10	27.57
C5 -420 F	44.59	43.39
420-700 F	24.23	23.66
700-END PT	5.07	5.38
C5+-END PT	73.90	72.43
ISO/NORMAL MOLE RATIO		
C4	0.0454	0.0416
C5	0.0799	0.0690
C6	0.1435	0.1493
C4=	0.0888	0.0683
PARAFFIN/OLEFIN RATIO		
C3	1.0302	1.0408
C4	0.7014	0.5102
C5	0.4578	0.4923
SCHULZ-FLORY DISTRBTN		
ALPHA (EXP(SLOPE))	0.8274	0.8275
RATIO CH4/(i-A)**2	5.0670	5.1144
ALPHA FRM CORRELATION		
ALPHA (EXPTL/CORR)	0.8537	0.8537
ALPHA (EXPTL/CORR)	0.9692	0.9693
W%CH4 FRM CORRELATION		
W%CH4 (EXPTL/CORR)	13.6196	13.8172
W%CH4 (EXPTL/CORR)	1.1078	1.1017
LIQ HC COLLECTION		
PHYS. APPEARANCE	GRN WAXY	GRN WAXY
DENSITY	0.748	0.754
N, REFRACTIVE INDEX	1.4285	1.4285
SIMULT'D DISTILATN		
10 WT % @ DEG F	265	277
16	303	304
50	446	451
84	644	648
90	696	706
RANGE(16-84 %)	341	344
WT % @ 420 F	44.00	43.50
WT % @ 700 F	90.31	89.54

NEW FORMAT JAN 25,85

#### IV. Run 3 (11723-16) with Catalyst 3 (Co/Th/X<sub>8</sub>/UCC-103+UCC-101)

This catalyst is the same as Catalyst 2 with two exceptions: the cobalt content is 75 percent of that of Catalyst 2, and the X<sub>8</sub> content is one percent of the cobalt content instead of 4 percent.

Conversion, product selectivity, isomerization of the pentane, and percent olefins of the C<sub>4</sub>'s are plotted against time on stream in Figs. A65-68. Simulated distillations of the C<sub>5</sub><sup>+</sup> product are plotted in Figs. A69-71. Carbon number product distributions are plotted in Figs. A72-78. Chromatograms from simulated distillations are reproduced in Figs. A79-81. Detailed material balances appear in Tables A9-10.

As compared with Catalyst 2 the initial activity was slightly lower, the conversion 10 percent lower, and the specific activity 25 percent lower. Per gram cobalt, therefore, the specific activity was the same for both.

The major difference between the two catalysts was in their water gas shift activities. With this catalyst the H<sub>2</sub>:CO usage ratio was greater than 2.0:1, only 6 percent of the oxygen having been rejected as CO<sub>2</sub>. With Catalyst 2 the water gas shift activity was higher: the H<sub>2</sub>:CO usage ratio less than 1.9:1, and about 11 percent of the oxygen rejected as CO<sub>2</sub>.

The conversion decreased at a rate of one percentage point

every 34 hours, as against every 48 hours for Catalyst 2; but with only 75 percent as much cobalt, the rate of decrease in specific activity was only a little higher. The conversion of H<sub>2</sub> decreased about four times faster than that of CO (one percentage point every 24 hours vs. every 97 hours, respectively), a pattern more characteristic of cobalt Fischer-Tropsch catalysts than that of Catalyst 2, with which the two rates were nearly equal. The faster loss of H<sub>2</sub> conversion resulted in decreasing water gas shift activity, as evinced by the decrease from 7.5 to 6.1 percent in the proportion of oxygen rejected as CO<sub>2</sub>.

The methane production, about 14 percent, was a little higher than with Catalyst 2 due mostly to differences between the H<sub>2</sub>:CO ratios in the reactors. The methane selectivity actually is more stable than that of Catalyst 2, increasing at a rate of one percentage point every 65 as against every 54 hours. Both effects are related to the fact that the methane production, initially about 15 percent more than the model predicts, soon stabilized, as with Catalyst 2, at the predicted level.

The production of C<sub>5</sub><sup>+</sup> was 73 percent initially and below 71 percent at the end of the run, a loss of one percentage point every 59 hours. This is lower in selectivity than Catalyst 2 (76 percent initially, more than 73 percent at a time on stream equivalent to this catalyst's end of run), and higher in stability (one percentage point every 48 hours). The C<sub>5</sub><sup>+</sup> mixes differed as well. With this catalyst most of the loss was in the diesel fraction, which decreased at a rate of one percentage point every

74 hours; both the gasoline fraction, and the ratio of gasoline to diesel, increased with time on stream. With Catalyst 2 the diesel production was steady, most of the loss was in heavies, and the gasoline to diesel ratio decreased.

There was a small degree of isomerization at first, but this quickly dropped to the usual low level. The level of olefins in the C<sub>4</sub> fraction was lower than with Catalyst 2. Except for the methane selectivity, the hydrocarbon distribution followed the Schulz-Flory distribution.

Ig appears to be a useful additive for intimately mixed catalysts, and at the level of 4 percent Ig to cobalt contributes both to stability and water gas shift activity. At lower levels (e.g., 1 percent Ig to cobalt), it helps with stability but not with water gas shift activity.

# RUN 11723-16

1:1 H<sub>2</sub>:CO  
890 PSIG  
880°C

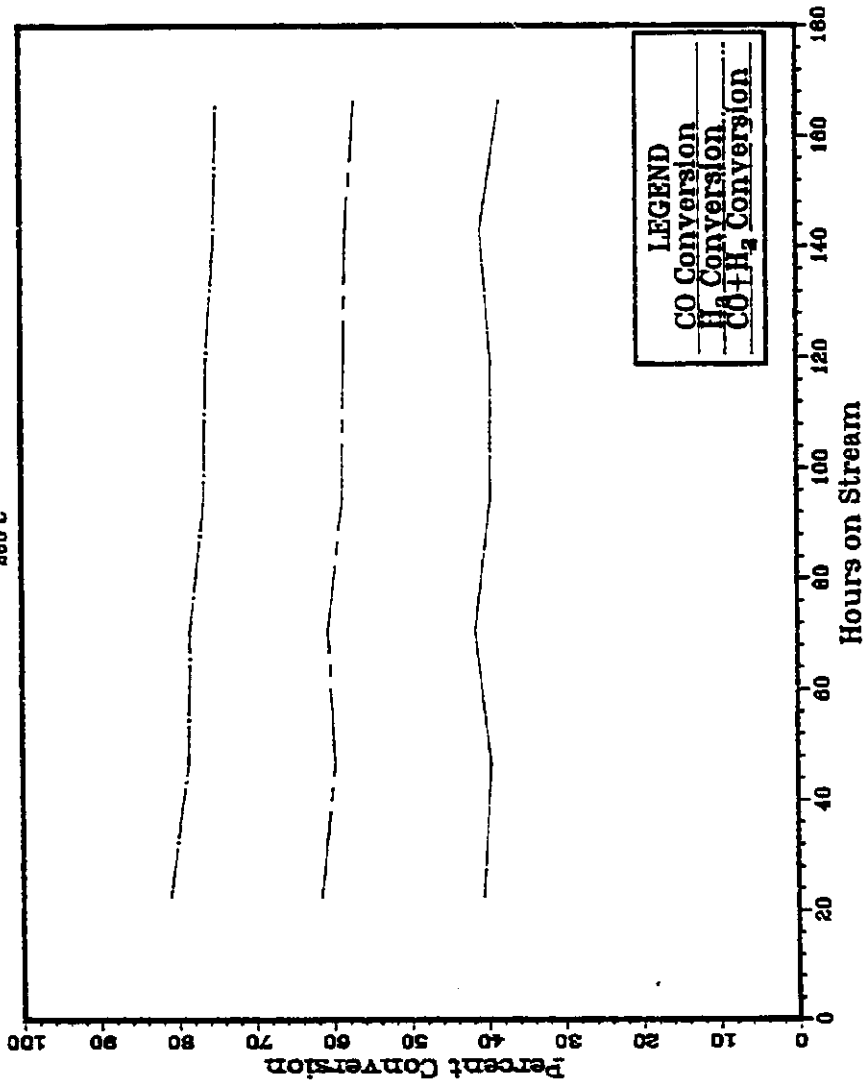


Fig. A65

# RUN 11723-16

1117.100  
 290 PSIG  
 880°C

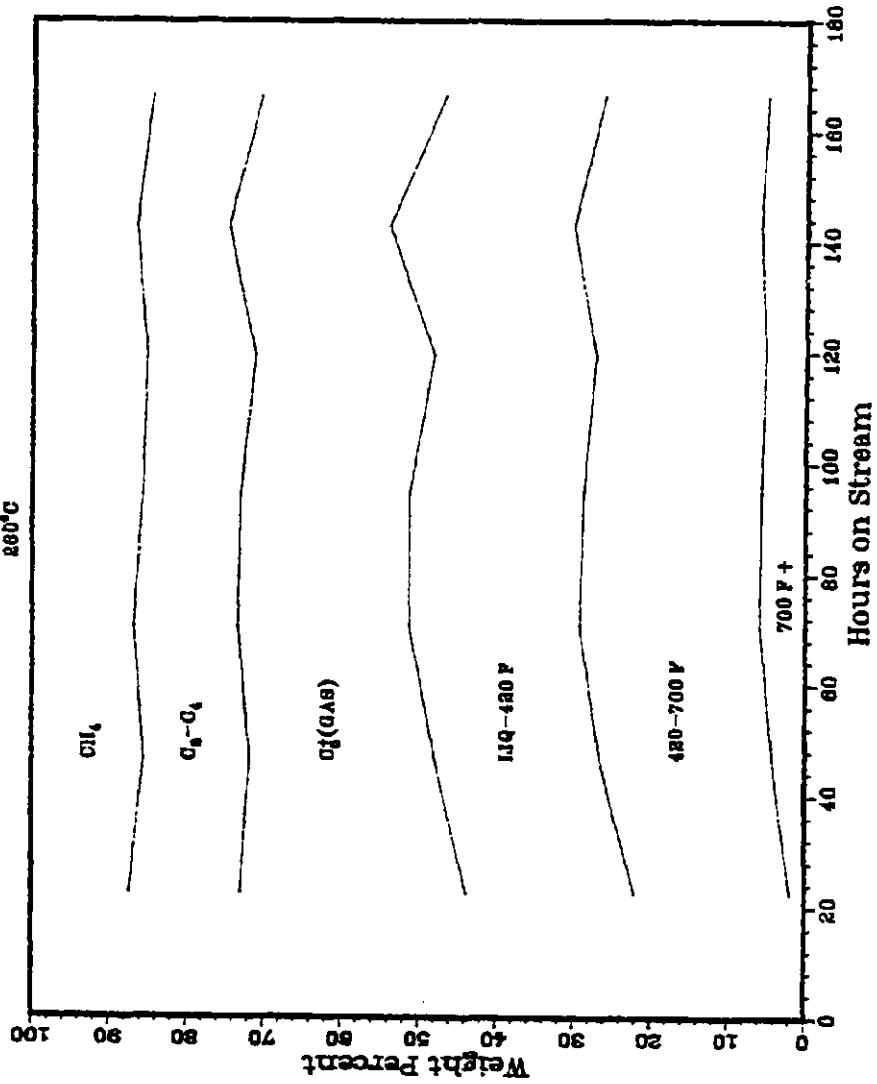


Fig. A66

RUN 11723-16

1:1 H<sub>2</sub>O  
800 PSIG  
260°C

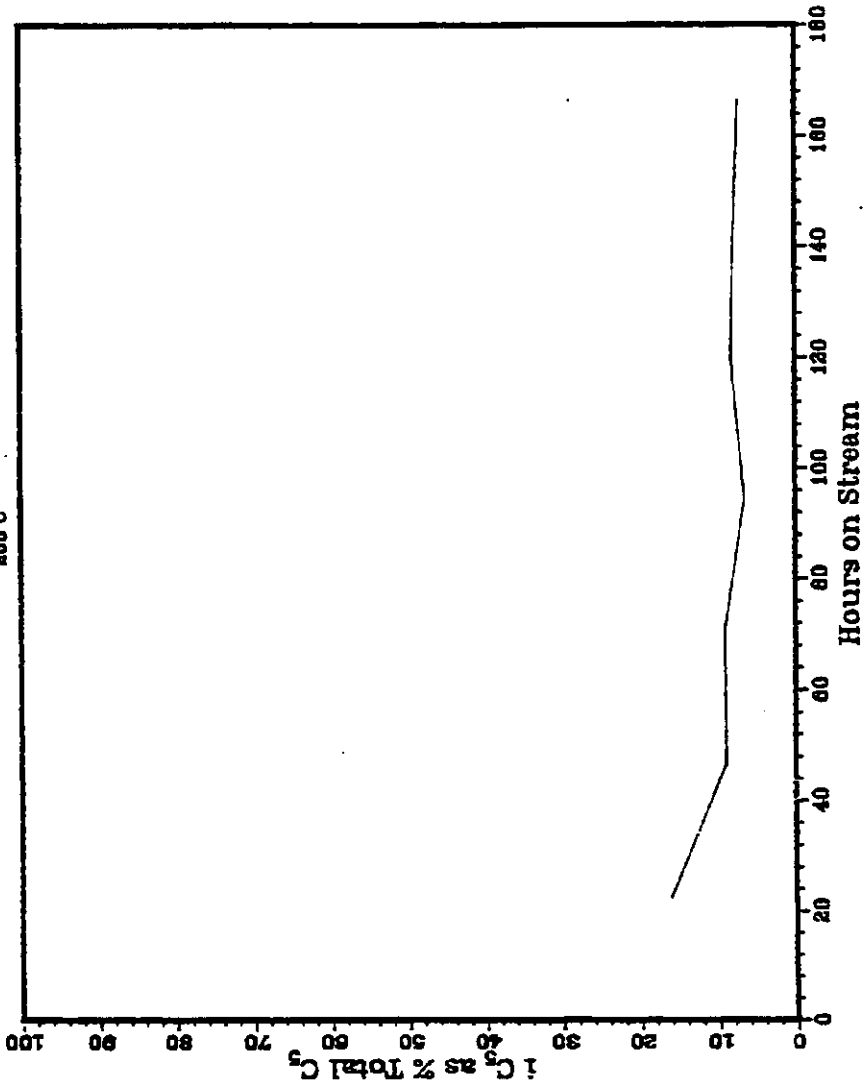


Fig. A67



# RUN 11723-16

111 H<sub>2</sub>O  
200 F810  
280°C

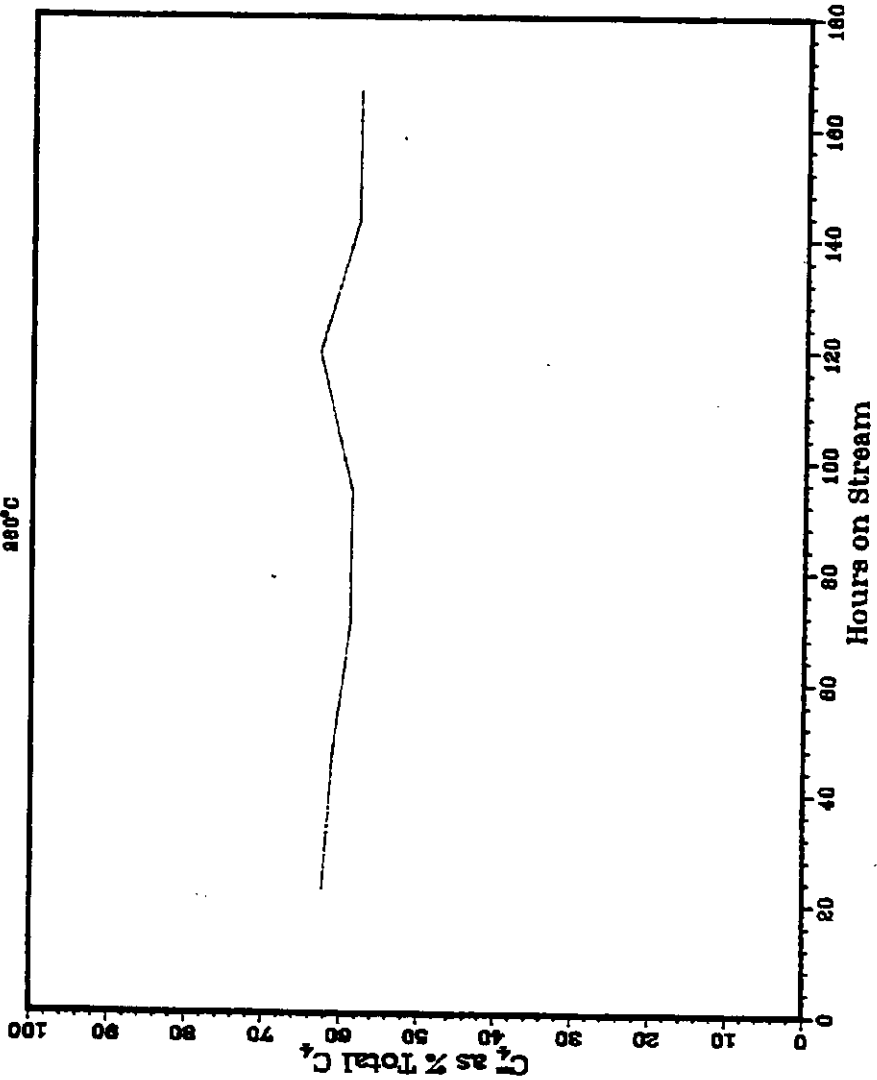


Fig. A68