



FE24676

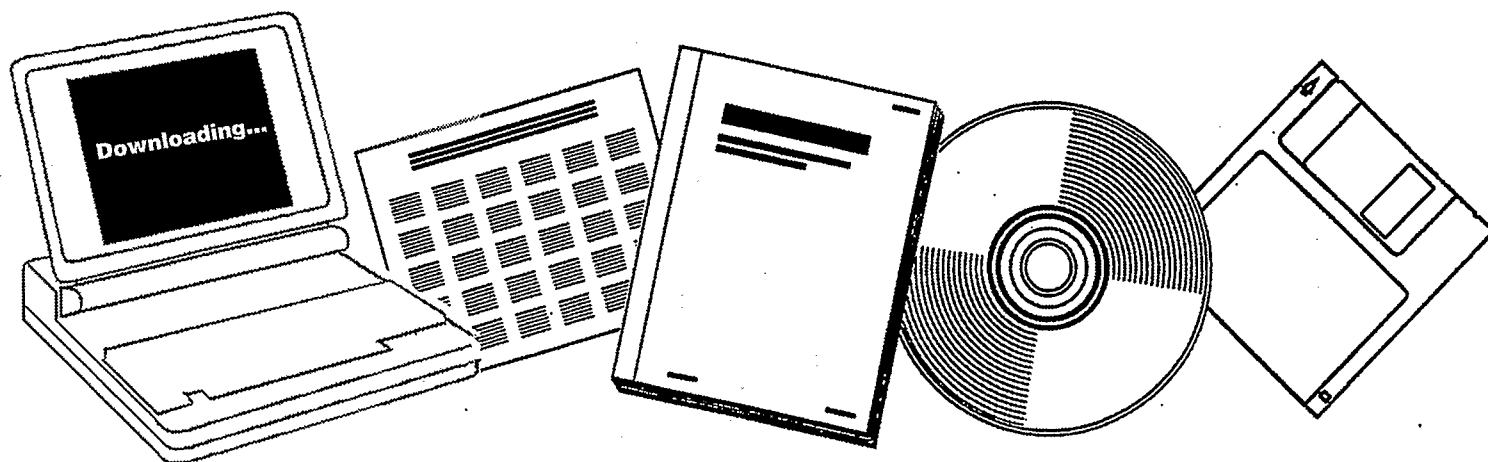
NTIS

One Source. One Search. One Solution

**TRANSITION METAL-GRAPHITE CATALYSTS FOR
PRODUCTION OF LIGHT HYDROCARBONS FROM
SYNTHESIS GAS. QUARTERLY REPORT, 1
NOVEMBER 1977--31 JANUARY 1978**

TEXAS A AND M UNIV., COLLEGE STATION.
DEPT. OF CHEMISTRY

FEB 1978



U.S. Department of Commerce
National Technical Information Service

One Source. One Search. One Solution.

NTIS



**Providing Permanent, Easy Access
to U.S. Government Information**

National Technical Information Service is the nation's largest repository and disseminator of government-initiated scientific, technical, engineering, and related business information. The NTIS collection includes almost 3,000,000 information products in a variety of formats: electronic download, online access, CD-ROM, magnetic tape, diskette, multimedia, microfiche and paper.



Search the NTIS Database from 1990 forward

NTIS has upgraded its bibliographic database system and has made all entries since 1990 searchable on **www.ntis.gov**. You now have access to information on more than 600,000 government research information products from this web site.

Link to Full Text Documents at Government Web Sites

Because many Government agencies have their most recent reports available on their own web site, we have added links directly to these reports. When available, you will see a link on the right side of the bibliographic screen.

Download Publications (1997 - Present)

NTIS can now provide the full text of reports as downloadable PDF files. This means that when an agency stops maintaining a report on the web, NTIS will offer a downloadable version. There is a nominal fee for each download for most publications.

For more information visit our website:

www.ntis.gov



U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Technical Information Service
Springfield, VA 22161

TRANSITION METAL-GRAPHITE CATALYSTS FOR PRODUCTION
OF LIGHT HYDROCARBONS FROM SYNTHESIS GAS

Quarterly Report for the Period
November 1, 1977 - January 31, 1978

Michael P. Rosynek

Texas A & M University
Department of Chemistry
College Station, Texas 77843

Date Submitted - February 1978

PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Under Contract No. E(49-18)-2467

RUSH

PRINTING TIME : Thu Dec 2 12:32:13 2004

ORDER : 1389394



PRODUCT : FE24676



LINE NUMBER : 0



MEDIA : BA



TITLE : TRANSITION METAL-GRAPHITE CATALYSTS FOR PRODUCTION OF LIGHT
HYDROCARBONS FROM SYNTHESIS GAS. QUARTERLY REPORT, 1
NOVEMBER 1977--31 JANUARY 1978

COPIES REQUESTED : 1

QUEUE : 13

JOB ID : 1037834

USER : RUSH

PAGE COUNT : 32

ABSTRACT

Cobalt-graphite intercalate, prepared by reduction of the corresponding CoCl_2 -graphite with an aromatic anion-radical, exhibits low to moderate activity for the hydrogenation of carbon monoxide to hydrocarbon products. Compared to a kieselguhr-supported cobalt catalyst, however, the graphite-based material produces substantially less methane (30-35% vs. 55-60% of all CO converted) and correspondingly more C_3^+ hydrocarbons (45-50% vs. 25-30% of all CO converted) at a reaction temperature of 250°C. The catalytic activity of cobalt-graphite is markedly dependent on the method of reduction of the CoCl_2 -graphite precursor. Reduction of CoCl_2 -graphite with sodium borohydride produces a cobalt-graphite intercalate that has much lower activity for carbon monoxide conversion than that prepared by reduction with an anion-radical.

I. OBJECTIVE AND SCOPE OF WORK

The objective of this research is the development of a novel process for the production of petrochemical feedstocks based on coal or other carbonaceous materials. Specifically, the project is to investigate the catalytic activities and selectivities of novel alkali and transition metal-graphites in producing light (C_1 - C_3) hydrocarbons from H_2/CO synthesis gas via the Fischer-Tropsch process.

II. SUMMARY OF PROGRESS TO DATE

A comparison of actual research progress to date vs. project schedule is contained in the "Project Plan and Progress Chart" shown in Fig. 1. In our previous Quarterly Report (for the period August 1, 1977 to October 31, 1977), we described comparative test results for the activity and product selectivity of a commercially-available cobalt/kieselguhr catalyst when used for the Fischer-Tropsch synthesis at one atmosphere pressure. During the most recent contract quarter, we have begun to evaluate the corresponding activity/selectivity characteristics of a cobalt-graphite intercalate for this reaction. In addition, we have briefly explored the effect on catalyst behavior of an alternative preparation method, and have completed the design, assembly, and installation of a microcomputer-based data processing system that will be employed during the current project. Technical details of research progress during the past quarter are described in the following sections.

III. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

A. Data Processing System

We have completed the fabrication and installation of a microcomputer-based data processing system that will be employed by the present project for on-site computation and tabulation of experimental reaction parameters and for processing gas chromatographic data during and after their experimental accumulation. The system is based on an IMSAI Corp. Model 8080 microcomputer and power supply, with 36K of user-accessible read/write memory, and is interfaced to a medium-speed digital tape system that is used for data/program storage and retrieval, and to a Teletype I/O device. Bootstrap routines and various tape operation instructions are contained in an additional 2K of read-only memory. Operating system software includes a relocatable assembler, monitor, and text editor and a tape control system.

Table I contains, for reference purposes, an "off-the-printer" listing of the program that is currently being employed for processing of Fischer-Tropsch reaction data, together with the complete output resulting from a sample set of data taken from a previous report. The program, requiring approximately 12K of core storage, was written for a MITS Inc. Extended

BASIC interpreter (Revision 4.1) that occupies an additional 14.6K of core, but could be used, following suitable minor modifications, with most high-level BASIC compilers. Much of the printed output shown in Table I is intended for interim monitoring purposes, and only the third page of output, containing product distribution, conversion, rate, and mass balance data, is normally provided in the report for any given experiment.

This system will permit considerably greater flexibility in monitoring the course of reaction during individual experiments than has existed previously, and will provide much greater versatility in computing and tabulating the results of each run.

B. Cobalt-Graphite

A commercially-available cobalt-graphite intercalate containing 3.4 wt% cobalt and prepared by anion-radical reduction of the corresponding CoCl_2 -graphite, was obtained from Alfa Chemicals Div. of Ventron Corp. for catalyst testing. Pretreatment of this material prior to all experiments involved contact with excess hydrogen for three hours at 300°C , followed by overnight evacuation at the same temperature, and final stabilization at the desired reaction temperature. All experiments were performed using the stirred-batch reactor system described in our first Quarterly Report.

1. Selectivity Characteristics

Tables II and III contain the results of two runs made at a reaction temperature of 250°C , each employing a separate and freshly-pretreated catalyst sample. Reproducibility, both in overall catalytic activity and in product distribution, between the two runs is not good, and it is not yet known which of the two sets of data more nearly represents the normal behavior of the cobalt-graphite intercalate. Subsequent discussion will focus on the results in Table III. For comparison purposes, Table IV presents the results for a corresponding run made at identical temperature, pressure, and pretreatment conditions, but in which a commercial kieselguhr-supported cobalt catalyst (containing 39 wt% Co) was employed.

Data is not yet available for the percentages of metal exposure that exist in the two types of catalysts, hence, relative activities of the two materials for CO conversion cannot be quantitatively compared. On an absolute basis, the rate of CO conversion (0.15 moles of CO/mole of Co/hr) over the cobalt-graphite intercalate was approximately 7 to 8 times slower than that over the supported cobalt catalyst. As in the case of the iron-graphites described previously, however, it is likely that this rate is considerably influenced by both the extent and the uniformity of cobalt intercalation, with "interior" cobalt atoms being essentially inaccessible as catalytic sites.

Of more interest, perhaps, is the considerable difference in the distribution of carbon-containing products that was observed for the two catalysts. At comparable extents of CO conversion (compare, for example,

the 4.00 hr sample in Table III with the 0.25 hr sample in Table IV), the cobalt-graphite catalyst produces considerably less methane than does the cobalt/kieselguhr (30-35% vs. 55-60% of all CO converted) and a correspondingly larger amount of C_3^+ hydrocarbons (45-50% vs. 25-30% of all CO converted). Extents of formation of carbon dioxide and of C_2 hydrocarbons, on the other hand, were virtually identical over the two catalysts. This selectivity behavior is analogous to that reported previously for an iron-graphite intercalate, which also generated less methane and more C_3^+ hydrocarbons than did an alumina-supported iron catalyst. Additional experiments, aimed at further characterizing these selectivity differences in the reaction temperature range 200-300°C, are currently in progress.

2. Effect of Preparation Method

Because of the irreproducibility of the first two runs made with the commercial cobalt-graphite intercalate, and in view of the relatively low overall activity for CO conversion exhibited in both cases, an alternative method of catalyst preparation was briefly explored. A $CoCl_2$ -graphite intercalate containing 12.7 wt% Co (obtained from Alfa Chemicals) was reduced by treatment with an excess of a 0.50 M aqueous solution of sodium borohydride for one hour at 100°C, followed by washing with de-ionized water and vacuum drying at 25°C. Assuming complete reduction of $CoCl_2$, the final product contained 6.2 wt% of cobalt. (Preparation of Ventron Corp. "Graphimet" metal intercalates, on the other hand, involves treatment of the corresponding metal chloride-graphite with a proprietary aromatic anion-radical reducing agent.)

Catalytic behavior of the borohydride-reduced material for CO hydrogenation at 300°C is compared with that of the cobalt/kieselguhr catalyst under identical reaction conditions in Tables V and VI. The overall initial activity of the borohydride-reduced cobalt-graphite catalyst for CO conversion was not only 200 times lower than that of the supported catalyst, but was also much lower, per cobalt atom, than that of the anion-radical-reduced material shown in Tables II and III (at 250°C). These results again suggest the relative inaccessibility of a large fraction of the cobalt atoms in the "interior" of graphite crystallites or, possibly, incomplete removal of excess $NaBH_4$ or reduction byproducts. Despite the very low activity exhibited by the borohydride-reduced cobalt-graphite, its tendency, even at a reaction temperature of 300°C, to produce considerably larger amounts of C_2^+ hydrocarbons, compared to the cobalt/kieselguhr catalyst, is again apparent, as can be seen by comparing the product distributions observed for the two catalysts.

In order to further examine the effect on catalytic properties of the borohydride reduction technique, an iron-graphite intercalate containing 1.9 wt% of Fe was prepared by reducing the corresponding $FeCl_3$ -graphite with $NaBH_4$ under conditions identical to those described above. Its catalytic behavior for CO hydrogenation at 300°C is compared to that of an iron-graphite "Graphimet" in Tables VII and VIII. It is evident that only slight differences, in both overall activity and in product distribution, exist

between the two iron-graphite materials. The relative insensitivity of the catalytic properties of iron-graphite intercalates to the method of iron reduction, compared to their cobalt counterparts, may reflect fundamental differences in the uniformity, depth of penetration, and resulting accessibility of intercalated FeCl_3 vs. CoCl_2 . In particular, a much larger fraction of intercalated FeCl_3 may remain near the edges of the graphite crystallites following intercalation (thus being both more susceptible to complete reduction and to subsequent interaction with reactant CO molecules) than is the case for CoCl_2 , which may penetrate the graphite layers more deeply and/or more uniformly.

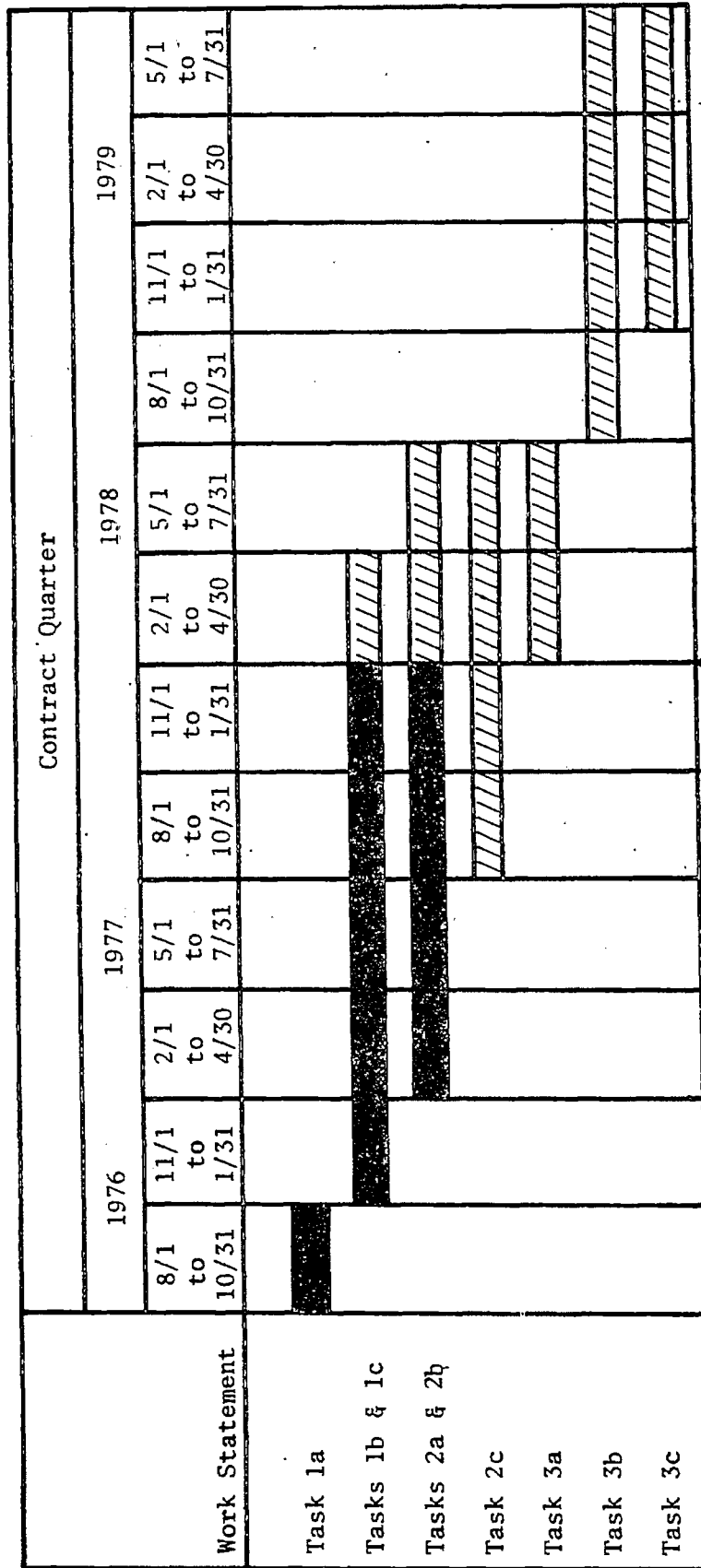
We do not plan to pursue further the applicability of the borohydride reduction method to the preparation of reduced metal-graphite intercalates, and will employ the commercially-available "Graphimets" for future studies of cobalt-graphite catalysts.

IV. CONCLUSIONS

Cobalt-graphite intercalate, prepared by reduction of the corresponding CoCl_2 -graphite with an aromatic anion-radical, exhibits low to moderate activity for the hydrogenation of carbon monoxide to hydrocarbon products. Compared to a kieselguhr-supported cobalt catalyst, however, the graphite-based material produces substantially less methane (30-35% vs. 55-60% of all CO converted) and correspondingly more C_3^+ hydrocarbons (45-50% vs. 25-30% of all CO converted) at a reaction temperature of 250°C. The catalytic activity of cobalt-graphite is markedly dependent on the method of reduction of the CoCl_2 -precursor. Reduction of CoCl_2 -graphite with sodium borohydride produces a cobalt-graphite intercalate that has much lower activity for carbon monoxide conversion than that prepared by reduction with an anion-radical.

FIGURE 1

Project Plan and Progress Chart



LEGEND



Scheduled



Completed



End of Reporting Period

Table I

```

1000 REM - PROGRAM FOR PROCESSING FISCHER-TROPSCH REACTION DATA
1010 REM -----
1020 REM
1030 REM - DEFINED STRING VARIABLES
1040 REM
1050 Z0$ = "EXPT. NO. - "
1060 Z1$ = "CATALYST - "
1070 Z2$ = "WEIGHT - "
1080 Z3$ = "REACTION TEMPERATURE = ### C"
1090 Z4$ = "INITIAL TOTAL PRESSURE = ### TORR (### ATM)"
1100 Z5$ = "INITIAL H2/CO RATIO = ###"
1110 Z6$ = "REACTOR VOLUME = 339 CC"
1120 Z7$ = "ROOM TEMPERATURE = ### C"
1130 Z8$ = "INITIAL H2 PRESSURE = ###.## TORR (### ATM)"
1140 Z9$ = "INITIAL CO PRESSURE = ###.## TORR (### ATM)"
1150 ZAS = "REXN TIME"
1160 ZBS = "      CO      CO2      CH4      C2H4      C2H6      C3H6      C3H8"
1170 ZCS = "      C4'S      C5+"
1180 ZDS = "-----"
1190 ZES = "-----"
1200 ZFS = "#####"
1210 ZGS = "-----"
1220 ZHS = "#####"
1230 ZIS = "FISCHER-TROPSCH REACTION RESULTS"
1240 ZJS = "-----"
1250 ZKS = "-----"
1260 ZLS = "-----"
1270 ZMS = "###.##"
1280 ZNS = "#####"
1290 ZOS = "#####"
1300 ZPS = "###.##"
1310 ZQS = "###.##"
1320 ZRS = "###.##"
1330 ZSS = "###.##"
1340 ZTS = "###.##"
1350 ZUS = "EXPT DATE - "
1360 REM
1370 REM - READ AND PRINT RAW DATA
1380 REM
1390 DEFINT I-N
1400 READ DT$, EX$, C1$, C2$, C3$, WT$, PH, PC, T1, T2, N
1410 DIM AX(N,8), BX(N,8), CX(N,8), PX(N,8), CB(N), CC(N), RA(N),
      SM(N), SU(N), TI(N), TT(N)
1420 READ AX(0,0)
1430 TI(0) = 01
1440 FOR I = 1 TO N
1450 READ TI(I), AX(I,0), AX(I,2), AX(I,1), AX(I,3), AX(I,4), AX(I,5),
      AX(I,6), AX(I,8), AX(I,7)
1460 NEXT I
1470 CA = AX(0,0) * .939

```

Table I (cont.)

```

1480 AI = CA/42
1490 CM = (PC * 5086.7)/(T1 + 273.15)
1500 PH = PH * 317/339
1510 PC = PC * 317/339
1520 PP = PH/PC
1530 H2 = PH/760
1540 TP = PH + PC
1550 CO = PC/760
1560 PT = TP/760
1570 M = 1
1580 PRINT TAB(32); "RAW DATA"
1590 PRINT TAB(32); "-----"
1600 PRINT
1610 PRINT ZUS; DTS : PRINT
1620 PRINT ZOS; EX$ : PRINT
1630 PRINT ZIS; C1$ : PRINT TAB(13); C2$ : PRINT TAB(13); C3$
1640 PRINT
1650 PRINT Z2$; WT$ : PRINT
1660 PRINT USING Z3$; T2
1670 PRINT USING Z4$; TP, PT
1680 PRINT USING Z5$; PP
1690 PRINT Z6$ : PRINT
1700 ON M GOTO 1710, 1940, 2420
1710 PRINT USING Z7$; T1
1720 PRINT USING Z8$; PH, H2
1730 PRINT USING Z9$; PC, CO
1740 PRINT : PRINT : PRINT
1750 PRINT TAB(35); "RAW PEAK AREAS"
1760 PRINT ZAS : PRINT " (MIN)";
1770 PRINT ZBS; ZCS
1780 PRINT ZDS; ZES : PRINT
1790 PRINT USING ZFS; TI(0), AX(0,0);
1800 PRINT ZGS : PRINT
1810 FOR I = 1 TO N
1820 PRINT USING ZFS; TI(1), AX(1,0);
1830 PRINT USING ZHS; AX(1,1), AX(1,2), AX(1,3), AX(1,4), AX(1,5),
      AX(1,6), AX(1,7), AX(1,8)
1840 PRINT
1850 NEXT I
1860 FOR I = 1 TO 10 : PRINT : NEXT I
1870 REM
1880 REM - CALCULATE MICROMOLES AND RATES OF CHANGE OF EACH COMPONENT
1890 REM
1900 AX(0,0) = CA
1910 PRINT TAB(20); ZIS : PRINT TAB(20); ZJS : PRINT
1920 M = 2
1930 GOTO 1620
1940 PRINT ZKS; ZLS : PRINT
1950 PRINT TAB(27); "MICROMOLES OF EACH COMPONENT"
1960 PRINT ZAS : PRINT " (HRS)"; ZBS; ZCS

```

Table I (cont.)

```

1970 PRINT ZDS; ZES : PRINT
1980 IF K = 2 GOTO 2200
1990 B = 11
2000 FOR I = 0 TO N
2010 F = B * CM/ΔI
2020 AX(I,0) = (AX(I,0) * F)/42!
2030 AX(I,1) = (AX(I,1) * F)/48!
2040 AX(I,2) = (AX(I,2) * F)/35.7
2050 AX(I,3) = (AX(I,3) * F)/48!
2060 AX(I,4) = (AX(I,4) * F)/51.2
2070 AX(I,5) = (AX(I,5) * F)/64.5
2080 AX(I,6) = (AX(I,6) * F)/64.5
2090 AX(I,7) = (AX(I,7) * F)/85!
2100 AX(I,8) = (AX(I,8) * F)/102!
2110 TI(I) = TI(I)/60
2120 PRINT USING ZMS; TI(I), AX(I,0);
2130 PRINT USING ZNS; AX(I,1), AX(I,2), AX(I,3), AX(I,4), AX(I,5),
      AX(I,6), AX(I,7), AX(I,8)
2140 B = B + 4.5E-03
2150 NEXT I
2160 PRINT : PRINT
2170 PRINT TAB(25); "COMPONENTS RATE OF CHANGE (%/HR)"
2180 K = 2
2190 GOTO 1960
2200 PX(I,0) = (100 * (AX(I,0) - AX(0,0)))/(AX(0,0) * TI(I))
2210 PRINT USING ZMS; TI(I), PX(I,0);
2220 PRINT ZGS
2230 FOR I = 2 TO N
2240 FOR J = 0 TO 8
2250 IF AX(I-1,J) <= 0 THEN GOTO 2260 ELSE GOTO 2280
2260 PX(I,J) = 0!
2270 GOTO 2290
2280 PX(I,J) = (100 * (AX(I,J) - AX(I-1,J)))/(AX(I-1,J) *
      (TI(I) - TI(I-1)))
2290 NEXT J
2300 PRINT USING ZMS; TI(I), PX(I,0);
2310 FOR J = 1 TO 8
2320 PRINT USING ZNS; PX(I,J);
2330 NEXT J,I
2340 FOR I = 1 TO 11 : PRINT : NEXT I
2350 STOP
2360 REM
2370 REM - COMPUTE MOLE PERCENTS AND CONVERSION PARAMETERS
2380 REM
2390 M = 3
2400 PRINT TAB(20); ZIS : PRINT TAB(20); ZJS : PRINT
2410 GOTO 1620
2420 PRINT ZKS; ZLS : PRINT
2430 PRINT TAB(12);
2440 PRINT "- MOLE PERCENTS OF CARBON-CONTAINING ";

```

Table I (cont.)

```

2450 PRINT "PRODUCTS IN GAS PHASE -"
2460 PRINT "REACTION"
2470 PRINT "TIME (HRS)   CO2      CH4      C2H4      C2H6      C3H6      ";
2480 PRINT "C3H8      C4'S      C5+"
2490 PRINT "-----"
2500 PRINT "-----"
2510 PRINT
2520 TT(0) = AX(0,0)
2530 FOR I = 1 TO N
2540 TT(I) = AX(I,0) + AX(I,1) + AX(I,2) + AX(I,3)*2 + AX(I,4)*2 +
      AX(I,5)*3 + AX(I,6)*3 + AX(I,7)*4 + AX(I,8)*5
2550 CB(I) = (100 * TT(I))/CM
2560 CC(I) = (100 * (TT(I) - AX(I,0)))/CM
2570 SU(I) = (AX(I,1) + AX(I,2) + AX(I,3) + AX(I,4) + AX(I,5) +
      AX(I,6) + AX(I,7) + AX(I,8))/100
2580 RA(I) = ((TT(I) - AX(I,0)) - (TT(I-1) - AX(I-1,0)))/
      (TI(I) - TI(I-1))
2590 FOR J = 1 TO 8
2600 BX(I,J) = AX(I,J)/SU(I)
2610 NEXT J
2620 PRINT USING ZOS; TI(I), BX(I,1);
2630 FOR J = 2 TO 8
2640 PRINT USING ZPS; BX(I,J);
2650 NEXT J,I
2660 PRINT : PRINT : PRINT
2670 PRINT "      ---- MOLES PER 100 MOLES OF CO CONVERTED ----"
2680 PRINT "REXN"; TAB(62); "RATE % C"
2690 PRINT "TIME   CO2   CH4   C2H4   C2H6   C3H6   C3H8   C4'S   C5+      ";
2700 PRINT "% CO MMOL MASS"
2710 PRINT "(HR)   (X1)  (X1)  (X2)  (X2)  (X3)  (X3)  (X4)  (X5)";
2720 PRINT "  CONV /HR  BAL."
2730 PRINT "----";
2740 FOR I = 1 TO 8 : PRINT " ----"; : NEXT I
2750 PRINT " ----" : PRINT
2760 FOR I = 1 TO N
2770 SM(I) = BX(I,1) + BX(I,2) + BX(I,3)*2 + BX(I,4)*2 + BX(I,5)*3
      + BX(I,6)*3 + BX(I,7)*4 + BX(I,8)*5
2780 G = 100/SM(I)
2790 CX(I,1) = BX(I,1) * G
2800 CX(I,2) = BX(I,2) * G
2810 CX(I,3) = BX(I,3) * G * 2
2820 CX(I,4) = BX(I,4) * G * 2
2830 CX(I,5) = BX(I,5) * G * 3
2840 CX(I,6) = BX(I,6) * G * 3
2850 CX(I,7) = BX(I,7) * G * 4
2860 CX(I,8) = BX(I,8) * G * 5
2870 IF TI(I) >= 10 THEN GOTO 2900 ELSE GOTO 2880
2880 PRINT USING ZQS; TI(I);
2890 GOTO 2910
2900 PRINT USING ZRS; TI(I);

```

Table I (cont.)

```

2910 FOR J = 1 TO 8
2920 PRINT USING ZS$; CX(I,J);
2930 NEXT J
2940 PRINT USING ZT$; CC(I), RA(I), CB(I)
2950 NEXT I
2960 FOR I = 1 TO 12 : PRINT : NEXT I
2970 STOP
2980 REM
2990 REM - PLOT CONVERSION AND PRODUCT DISTRIBUTION DATA
3000 REM
3010 AS$ = "##.##          ##.##          ##.##          ##.##"
3020 BS$ = "##.##          ##.##"
3030 CS$ = "TIME AXIS = ##.## HRS/DIVISION"
3040 DS$ = "LAST TIME = ##.## HRS"
3050 PRINT TAB(25); "PERCENT CO CONVERSION"
3060 PRINT : PRINT "0"; TAB(10);
3070 PRINT USING AS$; CC(N)/6, 2*CC(N)/6, 3*CC(N)/6, 4*CC(N)/6;
3080 PRINT USING BS$; 5*CC(N)/6, CC(N)
3090 PRINT "1"; TAB(12); "1"; TAB(24); "1"; TAB(36); "1"; TAB(48); "1";
3100 PRINT TAB(60); "1"; TAB(70); "1"
3110 FOR I = 1 TO 72 : PRINT "-"; : NEXT I : PRINT
3120 FOR I = 1 TO N
3130 Y = INT(45 * (TI(I) - TI(I-1))/TI(N) + .5)
3140 Z = INT(70 * CC(I)/CC(N) + .5)
3150 IF 40 * (TI(I) - TI(I-1))/TI(N) < .5 THEN GOTO 3260
      ELSE GOTO 3160
3160 IF Y < 3 THEN GOTO 3190 ELSE GOTO 3170
3170 FOR L = 1 TO Y-1
3180 PRINT "1"; TAB(71); "1" : NEXT L
3190 IF CC(I) > CC(N) THEN GOTO 3250 ELSE GOTO 3200
3200 IF CC(I) < 0 THEN GOTO 3230 ELSE GOTO 3210
3210 PRINT "1"; TAB(Z); "1"; TAB(71); "1"
3220 GOTO 3260
3230 PRINT "1 -"; TAB(71); "1"
3240 GOTO 3260
3250 PRINT "1 +"; TAB(71); "1"
3260 NEXT I
3270 PRINT "1"; TAB(71); "1"
3280 FOR I = 1 TO 72 : PRINT "-"; : NEXT I : PRINT
3290 PRINT : PRINT
3300 PRINT USING CS$; TI(N)/45
3310 PRINT USING DS$; TI(N)
3320 FOR I = 1 TO 12 : PRINT : NEXT I
3330 AS$ = "IIII TO **** = C02"
3340 BS$ = "***** TO XXXX = CH4"
3350 AC$ = "XXXX TO OOOO = C2'S"
3360 AD$ = "OOOO TO ++++ = C3'S"
3370 AE$ = "++++ TO IIII = C4'S"
3380 ES$ = "CONVERSION AXIS = ##.## PCT/DIVISION"
3390 FS$ = "LAST CONVERSION = ##.## PCT"

```

Table I (cont.)

```

3400 PRINT TAB(13); "CUMULATIVE MOLES PER 100 MOLES OF CO CONVERTED"
3410 PRINT
3420 PRINT "0"; TAB(15); "20"; TAB(29); "40"; TAB(43); "60";
3430 PRINT TAB(57); "80"; TAB(69); "100"
3440 PRINT "1"; TAB(15); "1"; TAB(29); "1"; TAB(43); "1";
3450 PRINT TAB(57); "1"; TAB(70); "1"
3460 FOR I = 1 TO 72 : PRINT "-"; : NEXT I : PRINT
3470 FOR I = 1 TO N
3480 A = INT(45 * (CC(I) - CC(I-1))/CC(N) + .5)
3490 B1 = INT(70 * CX(I,1)/100 + .5)
3500 B2 = INT(70 * (CX(I,1) + CX(I,2))/100 + .5)
3510 B3 = INT(70 * (CX(I,1) + CX(I,2) + CX(I,3) + CX(I,4))/100 + .5)
3520 B4 = INT(70 * (CX(I,1) + CX(I,2) + CX(I,3) + CX(I,4) + CX(I,5) +
      CX(I,6))/100 + .5)
3530 IF 40 * (CC(I) - CC(I-1))/CC(N) < .5 THEN GOTO 3590
      ELSE GOTO 3540
3540 IF A < 3 THEN GOTO 3570 ELSE GOTO 3550
3550 FOR L = 1 TO A-1
3560 PRINT "I"; TAB(71); "I" : NEXT L
3570 PRINT "I"; TAB(B1); "*"; TAB(B2); "X"; TAB(B3); "O";
3580 PRINT TAB(B4); "+"; TAB(71); "I"
3590 NEXT I
3600 PRINT "I"; TAB(71); "I"
3610 FOR I = 1 TO 72 : PRINT "-"; : NEXT I : PRINT
3620 PRINT : PRINT
3630 PRINT USING ES; CC(N)/45;
3640 PRINT TAB(50); A$
3650 PRINT TAB(50); AB$
3660 PRINT USING FS; CC(N);
3670 PRINT TAB(50); AC$
3680 PRINT TAB(50); AD$
3690 PRINT TAB(50); AE$
3700 FOR I = 1 TO 12 : PRINT : NEXT I
3710 PRINT FRE(0); " BYTES FREE IN B-28K VERSION"
3720 PRINT : PRINT
3730 END
3740 REM
3750 REM
3760 REM - DATA STATEMENTS START AT NUMBER 5000.
3770 REM
3780 REM - FIRST : EXPERIMENT DATE (IN QUOTES).
3790 REM - SECOND : EXPERIMENT RUN AND SERIES NUMBERS (IN QUOTES).
3800 REM - THIRD : FIRST LINE OF CATALYST DESCRIPTION (IN QUOTES).
3810 REM - FOURTH : SECOND LINE OF CATALYST DESCRIPTION (IN QUOTES).
3820 REM - FIFTH : THIRD LINE OF CATALYST DESCRIPTION (IN QUOTES).
3830 REM - SIXTH : CATALYST WEIGHT AND COMMENTS (ALL IN QUOTES).
3840 REM - SEVENTH : INITIAL H2 PRESSURE (IN TORR), INITIAL CO PRESSURE
3850 REM (IN TORR), ROOM TEMPERATURE (DEG C), REACTION TEMP-
3860 REM ERATURE (DEG C), TOTAL NUMBER OF SAMPLES (NOT IN-
3870 REM CLUDING T = 0).

```


Table I (cont.)

```

3880 REM - EIGHTH : AREA OF CO PEAK AT T = 0.
3890 REM - REST : REACTION TIME (IN MINUTES), FOLLOWED BY PEAK AREAS
3900 REM OF CO, CH4, CO2, C2H4, C2H6, C3H6, C3H8, C5+, AND
3910 REM C4'S, IN THAT ORDER.
3920 REM
3930 REM
3940 REM - BEGINNING OF DATA STATEMENTS
3950 REM
5000 DATA "NOVEMBER 13, 1977"
5010 DATA "SERIES 2, RUN 1-A"
5020 DATA "COBALT ON KIESELGUHR, DOUBLY-PROMOTED (CO : THO2 : MGO : "
5030 DATA "KIESELGUHR = 31.4 : 1.9 : 3.8 : 63.0 WT%); REDUCED IN H2"
5040 DATA "FOR 3 HRS AT 400 C, THEN EVACUATED FOR 16 HRS AT 300 C."
5050 DATA "0.01 G TOTAL"
5060 DATA 500, 250, 26.4, 225, 11
5070 DATA 730833
5080 DATA 15,668020,953,155,170,142,564,0,350,150
5090 DATA 30,647825,2045,242,222,370,926,0,300,479
5100 DATA 60,635807,4317,416,226,831,1700,456,2769,1714
5110 DATA 90,614573,6448,574,245,1237,2224,873,4371,3066
5120 DATA 120,590928,8518,727,250,1686,2624,1179,5583,3908
5130 DATA 180,555641,12581,1018,262,2499,3295,2126,7523,5406
5140 DATA 331,502125,21972,1695,268,4360,4174,4660,12143,9232
5150 DATA 420,465978,27052,2085,272,5338,4405,6100,13776,11095
5160 DATA 540,421535,33556,2589,300,6638,4489,7891,16794,13274
5170 DATA 983,314392,53823,4291,294,10573,4767,13781,24181,19907
5180 DATA 1080,296024,57366,4614,318,11183,4778,14852,25338,21333
OK

```

Table I (cont.)

RAW DATA

EXPT DATE - NOVEMBER 13, 1977

EXPT. NO. - SERIES 2, RUN 1-A

CATALYST - COBALT ON KIESELGUHR, DOUBLY-PROMOTED (CO : THO₂ : MGO :
KIESELGUHR = 31.4 : 1.9 : 3.8 : 63.0 WT%); REDUCED IN H₂
FOR 3 HRS AT 400 C, THEN EVACUATED FOR 16 HRS AT 300 C.

WEIGHT - 0.01 G TOTAL

REACTION TEMPERATURE = 225 C
INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)
INITIAL H₂/CO RATIO = 2.0
REACTOR VOLUME = 339 CC

ROOM TEMPERATURE = 26.4 C
INITIAL H₂ PRESSURE = 467.6 TORR (0.62 ATM)
INITIAL CO PRESSURE = 233.8 TORR (0.31 ATM)

RAW PEAK AREAS

REXN TIME (MIN)	CO	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.0	730833	--	--	--	--	--	--	--	--
15.0	668020	155	953	170	142	564	0	150	350
30.0	647825	242	2045	222	370	926	0	479	300
60.0	635807	416	4317	226	831	1700	456	1714	2769
90.0	614573	574	6448	245	1237	2224	873	3066	4371
120.0	590928	727	8518	250	1686	2624	1179	3908	5583
180.0	555641	1018	12581	262	2499	3295	2126	5406	7523
331.0	502125	1695	21972	268	4360	4174	4660	9232	12143
420.0	465978	2085	27052	272	5338	4405	6100	11095	13776
540.0	421535	2589	33556	300	6638	4489	7891	13274	16794
983.0	314392	4291	53823	294	10573	4767	13781	19907	24181
1080.0	296024	4614	57366	318	11183	4778	14852	21333	25338

Table I (cont.)

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 2, RUN 1-A

CATALYST - COBALT ON KIESELGUHR, DOUBLY-PROMOTED (CO : THO₂ : MGO :
KIESELGUHR = 31.4 : 1.9 : 3.8 : 63.0 WT%); REDUCED IN H₂
FOR 3 HRS AT 400 C, THEN EVACUATED FOR 16 HRS AT 300 C.

WEIGHT - 0.01 G TOTAL

REACTION TEMPERATURE = 225 C
INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)
INITIAL H₂/CO RATIO = 2.0
REACTOR VOLUME = 339 CC

MICROMOLES OF EACH COMPONENT

REXN TIME (HRS)	CO	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.00	4245.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.25	4151.1	0.8	7.0	0.9	0.7	2.3	0.0	0.5	0.9
0.50	4043.6	1.3	15.0	1.2	1.9	3.8	0.0	1.5	0.8
1.00	3986.3	2.3	31.8	1.2	4.3	6.9	1.9	5.3	7.1
1.50	3870.3	3.2	47.8	1.4	6.4	9.1	3.6	9.5	11.3
2.00	3737.8	4.0	63.4	1.4	8.7	10.8	4.9	12.2	14.5
3.00	3530.1	5.7	94.0	1.5	13.0	13.6	8.8	17.0	19.7
5.52	3204.1	9.5	164.9	1.5	22.8	17.3	19.4	29.1	31.9
7.00	2986.4	11.7	204.0	1.5	28.1	18.4	25.5	35.1	36.4
9.00	2713.3	14.6	254.1	1.7	35.0	18.8	33.1	42.2	44.5
16.38	2032.4	24.3	409.3	1.7	56.1	20.1	58.0	63.6	64.4
18.00	1921.9	26.2	438.2	1.8	59.6	20.2	62.8	68.4	67.7

COMPONENTS RATE OF CHANGE (%/HR)

REXN TIME (HRS)	CO	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.25	-8.9	--	--	--	--	--	--	--	--
0.50	-10.4	227.3	462.2	124.7	646.9	259.7	0.0	883.1	-55.6
1.00	-2.8	145.3	224.1	4.5	251.2	168.8	0.0	518.8	1654.2
1.50	-5.8	77.2	100.1	17.8	99.0	62.8	184.6	159.3	117.1
2.00	-6.8	54.4	65.4	5.0	73.8	37.0	71.3	56.1	56.6
3.00	-5.6	40.6	48.3	5.3	48.9	26.1	81.1	38.9	35.3
5.52	-3.7	26.7	30.0	1.1	29.9	10.8	47.7	28.4	24.7
7.00	-4.6	15.9	15.9	1.3	15.5	4.0	21.2	14.0	9.4
9.00	-4.6	12.4	12.3	5.4	12.4	1.2	15.0	10.1	11.2
16.38	-3.4	9.0	8.3	-0.2	8.1	0.9	10.2	6.9	6.0
18.00	-3.4	4.9	4.4	5.3	3.9	0.4	5.1	4.7	3.2

Table I (cont.)

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 2, RUN 1-A

CATALYST - COBALT ON KIESELGUHR, DOUBLY-PROMOTED (CO : THO₂ : MGO :
KIESELGUHR = 31.4 : 1.9 : 3.8 : 63.0 WT%); REDUCED IN H₂
FOR 3 HRS AT 400 C, THEN EVACUATED FOR 16 HRS AT 300 C.

WEIGHT - 0.01 G TOTAL

REACTION TEMPERATURE = 225 C

INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)

INITIAL H₂/CO RATIO = 2.0

REACTOR VOLUME = 339 CC

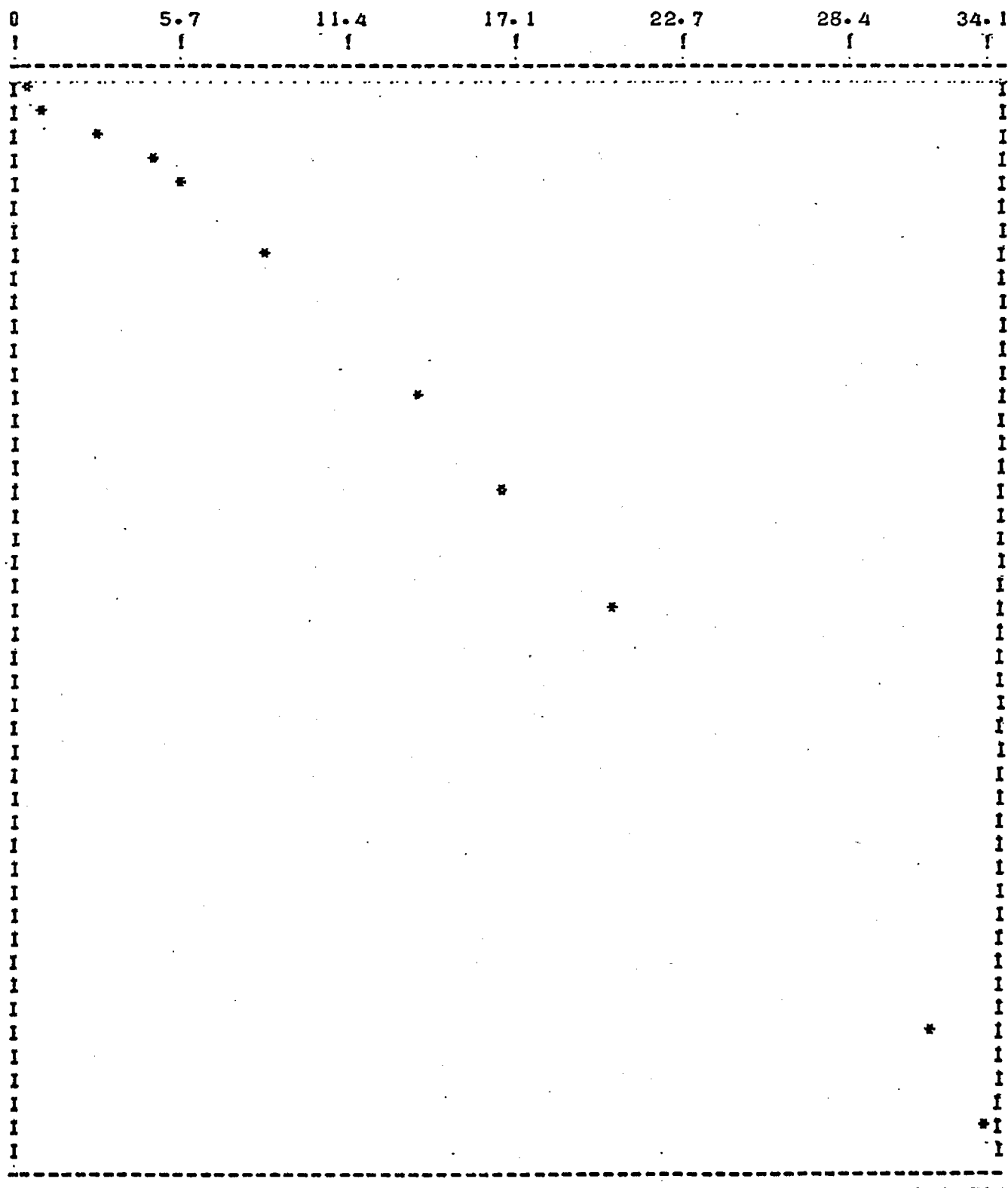
- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.25	6.4	53.2	7.1	5.5	17.4	0.0	3.5	6.8
0.50	5.2	59.0	4.8	7.4	14.8	0.0	5.8	3.0
1.00	3.7	52.3	2.0	7.0	11.4	3.1	8.7	11.7
1.50	3.4	51.8	1.5	6.9	9.9	3.9	10.3	12.3
2.00	3.4	52.8	1.2	7.3	9.0	4.0	10.2	12.1
3.00	3.3	54.3	0.8	7.5	7.9	5.1	9.8	11.4
5.52	3.2	55.6	0.5	7.7	5.9	6.5	9.8	10.8
7.00	3.2	56.6	0.4	7.8	5.1	7.1	9.7	10.1
9.00	3.3	57.2	0.4	7.9	4.2	7.4	9.5	10.0
16.38	3.5	58.7	0.2	8.0	2.9	8.3	9.1	9.2
18.00	3.5	58.8	0.2	8.0	2.7	8.4	9.2	9.1

----- MOLES PER 100 MOLES OF CO CONVERTED -----

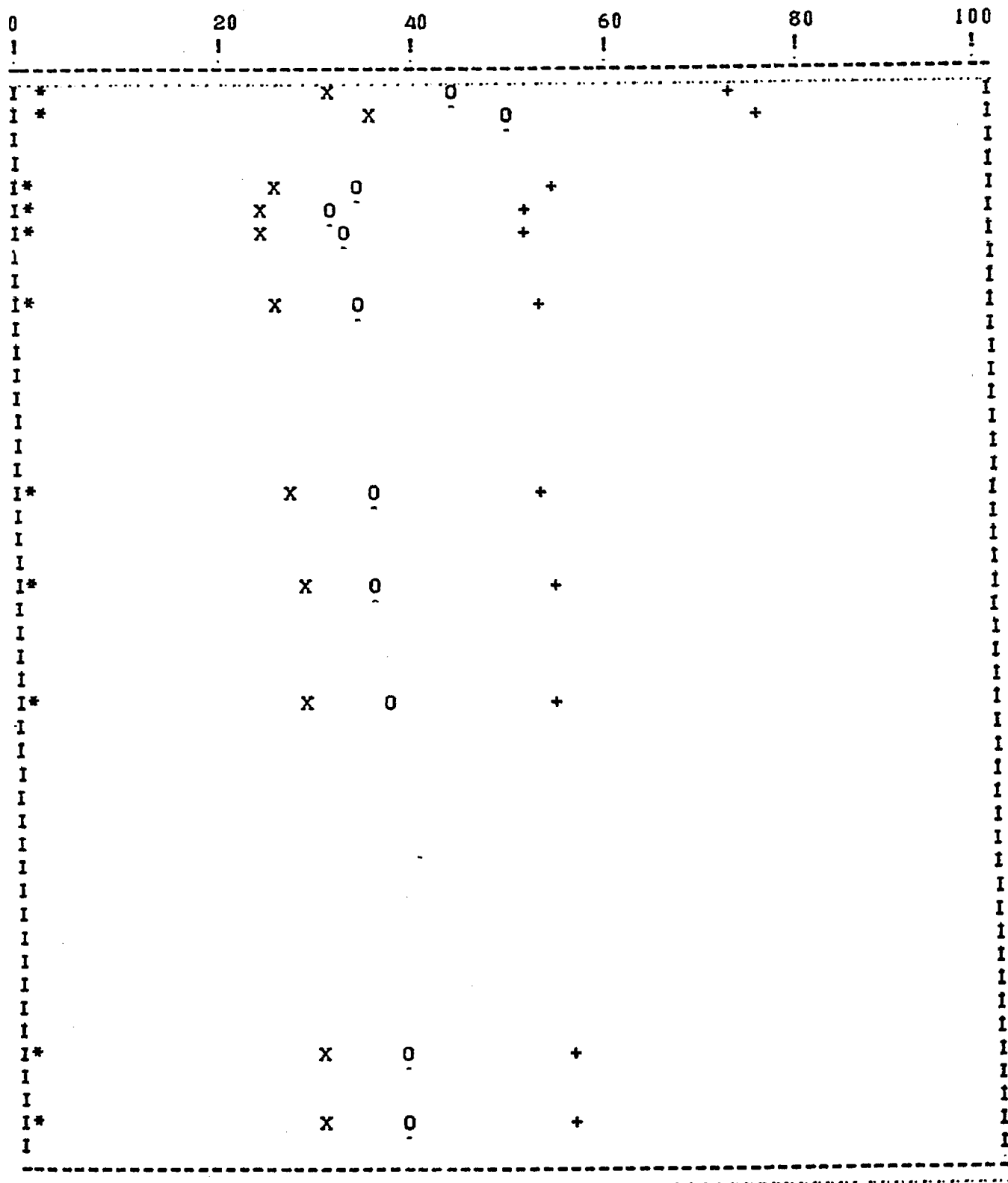
REXN TIME (HR)	CO ₂ (X1)	CH ₄ (X1)	C ₂ H ₄ (X2)	C ₂ H ₆ (X2)	C ₃ H ₆ (X3)	C ₃ H ₈ (X3)	C ₄ 'S (X4)	C ₅ +	% CO CONV	RATE MMOL /HR	% C MASS BAL.
0.25	3.5	28.7	7.6	6.0	28.2	0.0	7.6	18.4	0.6	97	98
0.50	3.0	34.4	5.6	8.7	25.9	0.0	13.6	8.8	1.0	77	96
1.00	1.8	24.8	1.9	6.6	16.2	4.3	16.5	27.8	3.0	170	97
1.50	1.6	24.0	1.4	6.4	13.7	5.4	19.1	28.4	4.7	142	96
2.00	1.6	24.7	1.1	6.8	12.7	5.7	19.1	28.4	6.0	114	94
3.00	1.6	26.0	0.8	7.2	11.3	7.3	18.7	27.2	8.5	106	92
5.52	1.6	27.1	0.5	7.5	8.5	9.5	19.1	26.2	14.3	98	90
7.00	1.6	28.0	0.4	7.7	7.6	10.5	19.3	24.9	17.2	81	88
9.00	1.6	28.6	0.4	7.9	6.3	11.2	19.0	25.0	20.9	80	85
16.4	1.8	30.1	0.2	8.2	4.4	12.8	18.7	23.7	32.0	64	80
18.0	1.8	30.2	0.2	8.2	4.2	13.0	18.9	23.4	34.1	55	79

PERCENT CO CONVERSION



TIME AXIS = 0.40 HRS/DIVISION
 LAST TIME = 18.00 HRS

CUMULATIVE MOLES PER 100 MOLES OF CO CONVERTED



CONVERSION AXIS = 0.76 PCT/DIVISION

LAST CONVERSION = 34.1 PCT

1111 TO **** = C02
 **** TO XXXX = CH4
 XXXX TO 0000 = C2'S
 0000 TO ++++ = C3'S
 ++++ TO 1111 = C4+

Table II

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 1, RUN 1-A

CATALYST - COBALT-GRAPHITE (3.4 WT% CO); VENTRON CORP. 'GRAPHIMET'
 NO. 89650; PRETREATED IN H₂ FOR 3 HRS AT 300 °C, THEN EVAC-
 UATED FOR 16 HRS AT 300 °C.

WEIGHT - 0.50 G TOTAL

REACTION TEMPERATURE = 251 °C
 INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)
 INITIAL H₂/CO RATIO = 2.0
 REACTOR VOLUME = 339 CC

- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
1.00	100.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
2.00	15.4	69.4	7.6	7.5	0.0	0.0	0.0	0.0
2.50	13.7	71.7	7.1	7.5	0.0	0.0	0.0	0.0
4.00	10.1	69.9	5.1	7.4	3.5	4.0	0.0	0.0
9.75	7.6	69.3	3.3	7.8	5.3	4.1	1.8	0.7
12.08	7.6	67.6	2.8	8.0	4.9	4.9	2.5	1.6
21.33	7.5	66.4	1.8	8.6	4.0	5.4	3.9	2.5

----- MOLES PER 100 MOLES OF CO CONVERTED -----

REXN TIME (HR)	CO ₂ (X1)	CH ₄ (X1)	C ₂ H ₄ (X2)	C ₂ H ₆ (X2)	C ₃ H ₆ (X3)	C ₃ H ₈ (X3)	C ₄ 'S (X4)	C ₅ +	% CO CONV	RATE MMOL /HR	% C MASS BAL.
1.00	100.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1	99
2.00	13.4	60.3	13.2	13.1	0.0	0.0	0.0	0.0	0.2	7	99
2.50	11.9	62.6	12.5	13.0	0.0	0.0	0.0	0.0	0.2	5	98
4.00	8.0	54.9	8.0	11.6	8.2	9.3	0.0	0.0	0.5	8	96
9.75	5.5	50.0	4.8	11.3	11.5	8.9	5.3	2.7	1.7	8	96
12.1	5.2	46.8	3.9	11.1	10.2	10.2	7.1	5.4	2.2	10	95
21.3	4.9	44.0	2.4	11.4	7.9	10.7	10.3	8.3	4.3	9	92

Table III

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 2, RUN 1-A

CATALYST - COBALT-GRAPHITE (3.4 WT% CO); VENTRON CORP. 'GRAPHIMET'
NO. 89650; PRETREATED IN H₂ FOR 3 HRS AT 300 °C, THEN EVAC-
UATED FOR 16 HRS AT 300 °C.

WEIGHT - 0.50 G TOTAL

REACTION TEMPERATURE = 250 °C
INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)
INITIAL H₂/CO RATIO = 2.0
REACTOR VOLUME = 339 CC

- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
1.00	6.1	57.5	3.8	9.0	6.6	5.7	6.9	4.4
2.00	5.7	60.1	2.8	9.6	5.3	7.0	5.5	3.9
3.00	5.3	59.0	2.1	9.7	4.8	8.2	6.5	4.3
4.00	5.2	59.9	1.7	10.2	4.1	8.7	5.8	4.4
5.00	5.1	59.3	1.4	10.3	4.3	8.9	5.9	4.9
12.50	5.4	58.8	0.6	11.0	2.0	10.7	6.5	5.1
24.00	6.0	57.7	0.3	11.3	1.2	11.7	6.6	5.3

----- MOLES PER 100 MOLES OF CO CONVERTED -----

REXN TIME (HR)	CO ₂ (X1)	CH ₄ (X1)	C ₂ H ₄ (X2)	C ₂ H ₆ (X2)	C ₃ H ₆ (X3)	C ₃ H ₈ (X3)	C ₄ 'S (X4)	C ₅ +	% CO CONV	RATE MMOL /HR	% C MASS BAL.
1.00	3.5	32.8	4.3	10.2	11.2	9.8	15.7	12.5	0.9	40	98
2.00	3.4	35.5	3.3	11.3	9.5	12.4	13.1	11.5	1.9	43	98
3.00	3.1	33.8	2.4	11.1	8.2	14.2	14.9	12.4	3.1	49	98
4.00	3.0	34.8	1.9	11.8	7.1	15.2	13.6	12.6	4.1	41	97
5.00	2.9	33.9	1.6	11.7	7.4	15.2	13.4	13.9	5.2	49	97
12.5	3.0	33.3	0.7	12.4	3.4	18.2	14.7	14.4	12.4	41	93
24.0	3.4	32.4	0.4	12.7	2.0	19.6	14.8	14.8	21.4	33	89

Table IV

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 3, RUN 1-A

CATALYST - COBALT ON KIESELGUHR (39 WT% CO); HARSHAW CHEMICAL CO. NO. CO-0127; PRETREATED IN HYDROGEN FOR 8 HRS AT 400 C, THEN EVACUATED FOR 16 HRS AT 300 C.

WEIGHT - 0.10 G TOTAL

REACTION TEMPERATURE = 250 C

INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)

INITIAL H₂/CO RATIO = 2.0

REACTOR VOLUME = 339 CC

- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.25	8.0	73.1	0.7	7.9	3.6	3.9	1.9	0.9
0.50	5.2	75.2	0.4	8.0	2.0	4.8	3.0	1.4
1.00	4.0	77.7	0.2	8.0	0.9	5.1	2.8	1.2
1.50	3.7	78.8	0.1	7.9	0.6	5.2	2.5	1.2
2.00	3.6	79.4	0.1	7.8	0.4	5.2	2.3	1.2
3.01	3.6	80.3	0.1	7.6	0.3	4.9	2.1	1.1
4.00	3.8	80.7	0.0	7.5	0.2	4.7	2.0	1.1

----- MOLES PER 100 MOLES OF CO CONVERTED -----

REXN TIME (HR)	CO ₂ (X1)	CH ₄ (X1)	C ₂ H ₄ (X2)	C ₂ H ₆ (X2)	C ₃ H ₆ (X3)	C ₃ H ₈ (X3)	C ₄ 'S (X4)	C ₅ +	% CO CONV	RATE MMOL /HR	% C MASS BAL.
0.25	6.0	54.9	1.1	11.8	8.2	8.7	5.8	3.5	4.4	748	100
0.50	3.8	55.0	0.5	11.7	4.4	10.6	8.9	5.1	9.0	783	96
1.00	3.0	58.2	0.3	12.0	2.1	11.5	8.5	4.5	16.7	664	95
1.50	2.8	59.6	0.2	12.0	1.3	11.9	7.6	4.6	23.7	592	95
2.00	2.7	60.6	0.1	11.9	1.0	11.8	7.1	4.7	29.8	525	95
3.01	2.8	62.4	0.1	11.9	0.6	11.4	6.6	4.2	39.8	424	96
4.00	3.0	63.2	0.1	11.8	0.5	11.0	6.3	4.1	47.1	314	96

Table V

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 1, RUN 1-A

CATALYST - COBALT-GRAPHITE (6.2 WT% CO); PREPARED BY REDUCTION OF
COCL₂-GRAPHITE WITH NABH₄ FOR 1 HR AT 100 C.; PRETREATED
IN H₂ FOR 3 HRS AT 300 C, THEN EVACUATED 16 HRS AT 300 C.

WEIGHT - 0.50 G TOTAL

REACTION TEMPERATURE = 300 C

INITIAL TOTAL PRESSURE = 702 TORR (0.92 ATM)

INITIAL H₂/CO RATIO = 2.0

REACTOR VOLUME = 339 CC

- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.25	44.3	46.4	4.9	4.4	0.0	0.0	0.0	0.0
0.50	30.2	58.6	4.9	6.3	0.0	0.0	0.0	0.0
1.00	17.9	65.0	3.6	7.8	2.8	2.8	0.0	0.0
1.50	13.9	69.0	2.6	8.4	3.2	3.0	0.0	0.0
2.00	12.0	72.2	2.0	8.5	2.2	3.0	0.0	0.0
3.00	10.2	72.6	1.4	9.1	1.6	4.0	1.1	0.0
4.00	9.9	73.4	0.9	9.0	1.2	4.8	0.8	0.0
5.00	10.6	73.9	0.5	8.9	0.9	4.2	0.6	0.3
21.00	15.7	71.8	0.1	7.7	0.1	3.7	0.7	0.3

----- MOLES PER 100 MOLES OF CO CONVERTED -----

REXN TIME (HR)	CO ₂ (X1)	CH ₄ (X1)	C ₂ H ₄ (X2)	C ₂ H ₆ (X2)	C ₃ H ₆ (X3)	C ₃ H ₈ (X3)	C ₄ 'S (X4)	C ₅ +	% CO CONV	RATE MMOL /HR	% C MASS BAL.
0.25	40.5	42.4	9.0	8.1	0.0	0.0	0.0	0.0	0.1	21	99
0.50	27.1	52.7	8.8	11.4	0.0	0.0	0.0	0.0	0.2	13	99
1.00	14.6	53.1	6.0	12.7	6.9	6.9	0.0	0.0	0.4	19	99
1.50	11.3	55.9	4.2	13.6	7.9	7.2	0.0	0.0	0.6	18	99
2.00	10.0	59.7	3.3	14.1	5.5	7.4	0.0	0.0	0.8	16	100
3.00	8.2	58.0	2.3	14.5	3.8	9.7	3.4	0.0	1.3	19	100
4.00	8.0	59.1	1.4	14.5	2.8	11.7	2.5	0.0	1.8	22	100
5.00	8.6	60.2	0.9	14.6	2.2	10.2	2.0	1.4	2.3	21	99
21.0	13.2	60.7	0.1	13.0	0.2	9.3	2.2	1.2	9.0	18	95

Table VI

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 7, RUN 1-A

CATALYST - COBALT ON KIESELGUHR (39 WT% CO); HARSHAW CHEMICAL CO. NO. CO-0127; PRETREATED IN HYDROGEN FOR 8 HRS AT 400 C, THEN EVACUATED FOR 16 HRS AT 300 C.

WEIGHT - 0.10 G TOTAL

REACTION TEMPERATURE = 300 C

INITIAL TOTAL PRESSURE = 702 TORR (0.92 ATM)

INITIAL H₂/CO RATIO = 2.0

REACTOR VOLUME = 339 CC

- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.25	6.4	90.3	0.1	2.9	0.1	0.3	0.0	0.0
0.52	6.8	89.7	0.0	2.9	0.1	0.4	0.0	0.0
1.00	7.9	88.4	0.1	3.1	0.1	0.4	0.0	0.0
1.50	8.8	87.0	0.1	3.2	0.2	0.5	0.1	0.0

- MOLES PER 100 MOLES OF CO CONVERTED -

REXN TIME (HR)	CO ₂ (X1)	CH ₄ (X1)	C ₂ H ₄ (X2)	C ₂ H ₆ (X2)	C ₃ H ₆ (X3)	C ₃ H ₈ (X3)	C ₄ 'S (X4)	C ₅ + (X5)	% CO CONV	RATE MMOL /HR	% C MASS BAL.
0.25	6.2	86.9	0.1	5.5	0.4	0.9	0.0	0.0	26.2	4444	98
0.52	6.6	86.4	0.1	5.7	0.2	1.1	0.0	0.0	40.1	2220	98
1.00	7.6	84.9	0.1	5.9	0.2	1.2	0.0	0.0	51.9	1034	97
1.50	8.4	82.8	0.3	6.1	0.6	1.4	0.3	0.1	56.6	398	97

Table VII

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 1, RUN 1-A

CATALYST - IRON-GRAPHITE (1.9 WT% FE); PREPARED BY REDUCTION OF FeCl_3 -GRAPHITE WITH NaBH_4 FOR 1 HR AT 100 C; PRETREATED IN H_2 FOR 3 HRS AT 300 C, THEN EVACUATED FOR 16 HRS AT 300 C.

WEIGHT - 0.50 G TOTAL

REACTION TEMPERATURE = 300 C
 INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)
 INITIAL H_2/CO RATIO = 2.0
 REACTOR VOLUME = 339 CC

- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO_2	CH_4	C_2H_4	C_2H_6	C_3H_6	C_3H_8	$\text{C}_4^+\text{'S}$	C_5^+
0.25	4.1	39.0	20.9	0.0	23.3	0.0	8.1	4.6
0.50	5.7	29.8	19.0	3.4	23.8	0.0	10.6	7.8
1.00	9.5	35.8	13.6	7.2	19.8	2.3	6.6	5.2
1.67	12.0	35.6	8.2	9.7	16.2	3.0	8.8	6.5
2.00	13.1	35.8	6.5	10.3	14.9	3.7	9.0	6.7
3.00	16.5	38.2	4.2	12.1	12.5	5.0	7.5	4.1
4.02	18.3	38.0	2.9	12.4	10.2	5.7	7.5	4.8
5.00	19.8	38.1	2.4	12.6	8.7	6.3	7.3	4.7
6.00	21.3	38.3	2.0	12.9	7.6	6.8	6.9	4.2

----- MOLES PER 100 MOLES OF CO CONVERTED -----

REXN TIME (HR)	CO_2 (X1)	CH_4 (X1)	C_2H_4 (X2)	C_2H_6 (X2)	C_3H_6 (X3)	C_3H_8 (X3)	$\text{C}_4^+\text{'S}$ (X4)	C_5^+ (X5)	% CO CONV	RATE MMOL /HR	% C MASS BAL.
0.25	1.9	18.6	19.9	0.0	33.3	0.0	15.4	10.8	0.5	93	98
0.50	2.4	12.8	16.3	2.9	30.6	0.0	18.3	16.7	1.6	186	97
1.00	4.6	17.4	13.3	7.0	28.9	3.4	12.9	12.6	3.8	184	96
1.67	5.7	17.1	7.8	9.3	23.3	4.4	16.8	15.5	7.8	254	95
2.00	6.3	17.2	6.2	9.9	21.5	5.4	17.3	16.1	9.6	240	94
3.00	8.7	20.1	4.4	12.7	19.7	7.9	15.8	10.7	12.8	137	93
4.02	9.7	20.1	3.1	13.2	16.2	9.1	15.8	12.8	16.8	164	91
5.00	10.7	20.5	2.5	13.6	14.1	10.2	15.7	12.7	19.5	120	91
6.00	11.7	21.2	2.2	14.2	12.6	11.2	15.2	11.7	21.4	81	91

Table VIII

FISCHER-TROPSCH REACTION RESULTS

EXPT. NO. - SERIES 18, RUN 1-A

CATALYST - IRON-GRAPHITE (2.2 WT% FE); VENTRON CORP. 'GRAPHIMET' NO. 89654; PRETREATED IN HYDROGEN FOR 3 HRS AT 300 C, THEN EVACUATED FOR 1 HR AT 300 C PRIOR TO USE.

WEIGHT - 0.50 G (AS GRAPHITE)

REACTION TEMPERATURE = 300 C
 INITIAL TOTAL PRESSURE = 701 TORR (0.92 ATM)
 INITIAL H₂/CO RATIO = 2.0
 REACTOR VOLUME = 339 CC

- MOLE PERCENTS OF CARBON-CONTAINING PRODUCTS IN GAS PHASE -

REACTION TIME (HRS)	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 'S	C ₅ +
0.25	6.3	34.4	17.9	3.1	19.6	0.0	10.1	8.6
0.50	7.4	35.9	15.8	4.6	17.9	1.6	10.3	6.5
0.75	8.8	36.5	13.9	5.7	17.2	2.0	10.0	6.0
1.00	10.1	37.4	12.8	6.6	16.6	2.0	9.0	5.5
1.50	12.1	38.5	11.0	7.7	15.9	2.3	7.9	4.7
2.00	13.7	38.7	9.9	8.3	14.9	2.5	7.8	4.3
3.00	16.7	38.3	8.7	8.8	13.8	2.7	7.3	3.8
4.00	18.8	37.7	8.0	9.0	12.8	2.6	7.2	3.9

----- MOLES PER 100 MOLES OF CO CONVERTED -----

REXN TIME (HR)	CO ₂ (X1)	CH ₄ (X1)	C ₂ H ₄ (X2)	C ₂ H ₆ (X2)	C ₃ H ₆ (X3)	C ₃ H ₈ (X3)	C ₄ 'S (X4)	C ₅ +	% CO CONV	RATE MMOL /HR	% C MASS BAL.
0.25	2.8	15.3	15.9	2.7	26.1	0.0	18.0	19.2	2.1	353	101
0.50	3.4	16.6	14.6	4.2	24.8	2.3	19.0	15.1	4.1	348	100
0.75	4.1	17.2	13.1	5.4	24.4	2.8	18.9	14.2	5.9	306	101
1.00	4.9	18.2	12.5	6.4	24.2	2.9	17.6	13.4	7.3	227	100
1.50	6.1	19.5	11.2	7.8	24.1	3.4	16.0	11.9	9.5	189	100
2.00	7.1	20.0	10.2	8.5	23.0	3.8	16.1	11.2	11.3	151	100
3.00	8.9	20.4	9.3	9.4	22.1	4.3	15.5	10.1	13.8	106	100
4.00	10.1	20.3	8.6	9.7	20.8	4.2	15.5	10.6	15.7	80	100

SATISFACTION GUARANTEED

NTIS strives to provide quality products, reliable service, and fast delivery. Please contact us for a replacement within 30 days if the item you receive is defective or if we have made an error in filling your order.

► **E-mail: info@ntis.gov**

► **Phone: 1-888-584-8332 or (703)605-6050**

Reproduced by NTIS

National Technical Information Service
Springfield, VA 22161

***This report was printed specifically for your order
from nearly 3 million titles available in our collection.***

For economy and efficiency, NTIS does not maintain stock of its vast collection of technical reports. Rather, most documents are custom reproduced for each order. Documents that are not in electronic format are reproduced from master archival copies and are the best possible reproductions available.

Occasionally, older master materials may reproduce portions of documents that are not fully legible. If you have questions concerning this document or any order you have placed with NTIS, please call our Customer Service Department at (703) 605-6050.

About NTIS

NTIS collects scientific, technical, engineering, and related business information – then organizes, maintains, and disseminates that information in a variety of formats – including electronic download, online access, CD-ROM, magnetic tape, diskette, multimedia, microfiche and paper.

The NTIS collection of nearly 3 million titles includes reports describing research conducted or sponsored by federal agencies and their contractors; statistical and business information; U.S. military publications; multimedia training products; computer software and electronic databases developed by federal agencies; and technical reports prepared by research organizations worldwide.

For more information about NTIS, visit our Web site at
<http://www.ntis.gov>.

NTIS

**Ensuring Permanent, Easy Access to
U.S. Government Information Assets**



U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Technical Information Service
Springfield, VA 22161 (703) 605-6000
