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FLASH HYDROPYROLYSIS OF COAL (FHP)

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September 15, 1977

Presentation by

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I. BNL Program Objectives

The overall objectives of the BNL program is to obtain process chemistry information required for the development of a Flash Hydropyrolysis Process for the conversion of coal to liquids and gaseous hydrocarbon products.

II. Rationale

The present studies concentrate on the rapid gas phase non-catalytic hydrogenation of a non-caking coal (lignite) maximizing liquid hydrocarbon yields for distillate fuels and chemical feedstock. The features of a non-catalytic process and direct hydrogenation in one step to liquid distillates tend to improve the efficiency and reduce capital and operating costs compared to other liquefaction processes such as Fischer-Tropsch.

The basis of the process is to rapidly heat pulverized lignite to elevated temperatures in a hydrogen atmosphere. This devolatilizes the coal and opens up molecular bonds in the coal structure subsequently forming smaller molecular fragments which add on hydrogen to form hydrocarbon products. Rapid cooling of the products prevents further degradation of the hydrocarbons to char.

For the development and design of a process it is necessary to determine the process chemistry in detail for a system simulating large scale process conditions. This includes the determination of the carbon conversion to products as a function of temperature, pressure, residence time, coal preparation, type of coal, feed gas composition, and gas to coal feed ratio. Product composition and material balance closures must be obtained for all the major elements in coal including C, H, O, S and N. The physical and chemical characteristics of the products must be determined and process kinetic data, flow sheet development and economic evaluations must be obtained. All this information then serves as a basis for a decision to move into the process demonstration unit (PDU) phase.

III. Experimental Approach

The process chemistry information is being obtained in a 1 in. i.d. straight downflow entrained tubular reactor. A highly instrumented tubular reactor was designed, constructed, and now is in operation at Brookhaven yielding data on a daily basis. The design characteristics of the experimental unit are as follows.

Reactor size	1 in. i.d. x 12 ft long (8 ft heated)
Material	Inconel 617
Coal flow capacity	up to 2 lb/hr (down to 0.1 lb/hr)
Max. run time	∿ 2 hrs (Hopper capacity ∿ 4 lb)
Hydrogen flow	up to 10 lb/hr (down to 0 flow)
Design pressure	up to 4000 psi
Design temperature	up to 850°C
Coal residence time	∿ 0.5 sec (min.)
	∿ 20 sec (max.) (free fall for 50 μ particles)

Instrumentation	full pressure, temperature, and flow rate remotely controlled
Analytical	On-line gas chromatograph measuring 10 components every 8 min. with integrator and direct readout and compositional analysis. H_2, CO, $(+N_2)$, H_2O, CO_2, CH_4, C_2H_6, C_6H_6, C_7H_8, C_8H_{10}, and total HC.
Sample taps	every 2 ft along reactor
Product collection	Trap - char
	Water condenser - heavy liquid HC
	Low temp. condenser - BTX
	Batch gas sampler - gas

Approximately 100 runs with dried and ground (150 μ and ≤ 3% free moisture) North Dakota lignite were made to date, scanning the process variables of pressure, temperature, flow rates of coal and hydrogen, and several coal residence times. Base line pyrolysis runs with inert helium gas were also made.

IV. Summary of Experimental Results to Date

All the run data obtained with the 1 in. tubular reactor are plotted in a series of curves attached to this report. The general conclusions to be drawn to date can be summarized as follows:

1. The major products formed are:

   Liquids: BTX (mainly benzene), heavier hydrocarbons ($\geq C_9$)

   Gases : Methane and ethane ($\leq C_5$)

   Carbon monoxide, (CO), and carbon dioxide (CO_2)

   Solids : Char

2. The yields of BTX tend to reach a maximum in the pressure range of 1500 psi, to 2000 psi and in the temperature range of 700 to 800°C. Maximum conversions of 10% ± 1% of the carbon in lignite feed are obtained. Above 800°C and below 700°C the BTX tends to decrease.

3. The heavier liquid hydrocarbons tend to follow the BTX yield and is of the same order of magnitude reaching maximum values of approximately 10%.

4. The liquid BTX and heavier hydrocarbon yields increase with pressure, at a constant temperature of 750°C, when the pressure is increased from 500 to 2000 psi. The liquid yield remains fairly constant above 2000 psi, up to 4000 psi, which is the highest pressure run to date. At 800°C, however, over the same pressure range, the liquid yields decrease above pressures of 1500 psi.

5. The heavier liquids have a 50% B.P. range of 79°C to 117°C due mainly to benzene. The remaining material is largely benzene, naphthalene and phenanthrene.

6. The methane and ethane gas yields continually increase with temperature at a given pressure up to the maximum run temperature to date of 815°C. The gas yield also increases with pressure at a given temperature. A maximum methane yield of 54% was obtained at 750°C and 4000 psi under which condition, the ethane yield was 14% and the BTX and liquid HC combined was 10%. The CO yield was 3.5% and the total yield was 81.4%.

7. The ethane yield rises to about 40% of the methane yield in

the temperature range of 750°C.

8. The total yield of CO and CO_2 and the CO/CO_2 ratio increases with increasing temperature. The carbon conversion to CO and CO_2 ranges between 5 and 10%.

9. The total carbon conversion to all useful liquids and gaseous carbonaceous products rises with increasing temperature from 40% at 700°C to over 60% at 2000 psi. At 750°C, the total conversion increases from 30% at 500 psi to over 70% at 4000 psi.

10. The pyrolysis of lignite in flowing inert helium, under similar reactor conditions as the hydrogen runs, gave 7 times less total hydrocarbon yield than in the presence of hydrogen.

11. The total yields increases with increasing residence time from 2.4 to 9.6 seconds at 750°C and 800°C and a pressure of 2500 psi. The main feature with increasing residence time at 800°C was that liquid yields decrease markedly. Therefore, higher cool down rates might be important.

12. Approximately 50% of the sulfur in the lignite stays with the char and the remainder leaves with the gaseous and liquid hydrocarbons. The average yields at 750°C between 1500 and 2500 psi are shown in the attached table.

V. Problem Areas

Modifications of the equipment since initial construction have taken place as follows:

1. The electrically heated Inconel 601 hydrogen preheater was replaced after 18 hours of operation at temperatures up to 900°C and pressures to 3000 psi because of a rupture due to hydrogen embrittlement.

2. The Instron vibratory feeder was replaced with a positive screw

feeder. The feeder works well and produces much more reliable results.

3. There is difficulty analyzing the heavier liquid hydrocarbons especially those boiling above 175°C in the chromatograph. Some oil product may be lost in the lead lines from the sample taps. More reliable results have been obtained condensing and separating the oils in a condenser and trap.

4. The coal feeder bin capacity was increased to 3 lbs to allow full 2 hour runs.

5. Improved sulfur analysis is being developed to obtain more detailed sulfur balances.

VI. Economic Analysis

A parametric evaluation of a full-scale flash hydropyrolysis plant to produce liquid and gaseous fuel and chemical feedstock was conducted. The study indicates that for the product distribution experimentally obtained (1) the overall conversion and thermal efficiency increases until the char balance point for hydrogen production is reached. Above this point the conversion remains fairly constant at a value of about 60% conversion, (2) the % DCF follows the same trend, flattening out at about 60%, (3) the % DCF increases with decreasing pressure due to the more costly higher pressure plant, and (4) the % DCF increases as more liquid BTX and less hydrocarbon gas is formed.

VII. Future Plans

The future work planned consists of the following:

1. Complete parametric process chemistry experiments
   a. pressure scan, 500 psi to 4000 psi
   b. complete temperature scan, 500°C to 850°C

2. Determine effect of residence time
    a. take analyses through taps along the length of the reactor at 2 ft intervals
    b. vary coal and hydrogen flow rate
3. Recycle char and ash
    a. add to coal feed
4. Investigate effect of feed gas composition
    a. methane
    b. CO and CO_2
    c. H_2O
5. Determine effect of coal preparation
    a. particle size
    b. effect of moisture
6. Determine effect of type of coal
    a. subbituminous
    b. bituminous
7. Determine effect of catalytic agents
    a. heterogeneous (isosynthesis)
    b. homogeneous
8. Convert unit to longer continuous operation
    a. lock hoppers
    b. gas recirculation
9. Run cold flow experiments for reactor design

Reports Published to Date

T. V. Sheehan, Meyer Steinberg, and Quinton Lee, "Centrifugal coal flash hydrogenation in a rotating fluidized bed reactor for a full sized coal liquefaction plant", BNL 18268 (August 1973).

Meyer Steinberg and Peter Fallon, "Coal liquefaction by rapid gas phase hydrogenation", ACS Symposium Series, No. 20, Hydrocracking and Hydrotreating, American Chemical Society, pp. 123-135 (1976), and BNL 19507 (Nov. 1974).

Meyer Steinberg, T. V. Sheehan, and Quinton Lee, "Flash Hydropyrolysis Process for conversion of lignite to liquid and gaseous products", presented at ACS National Meeting, Div. of I&EC, NY, NY, April 4-9, 1976, Synthetic Fuels Processing Comparative Economics, Edited by Arnold N. Pelofsky, Chapter VIII, pp. 163-192, Marcel Dekker, Inc., New York (1977), and BNL 20915 (January 1976).

Peter Fallon and Meyer Steinberg, "Flash Hydropyrolysis of Coal-- the design, construction, operation and initial results of a Flash Hydropyrolysis Experimental Unit", ACS 173rd Nat. Mtg., New Orleans, La., and BNL 22519 (March 1977).

Peter Fallon and Meyer Steinberg. "Flash Hydropyrolysis of Coal, Quarterly Report No. 1", to FE/ERDA, BNL 50677 (June 1977).

Meyer Steinberg and Peter Fallon, "Flash Hydropyrolysis of Coal, Quarterly Report No. 2", (August 1977).

Monthly letter reports were issued starting in May 1975 and have run consecutively through July 1977.

B. Bhatt, Peter Fallon and Meyer Steinberg, "Economic Analysis of Flash Hydropyrolysis", BNL 22912 (May 1977).

BNL PROGRAM OBJECTIVES

FLASH HYDROPYROLYSIS OF COAL

1. OBTAIN PROCESS CHEMISTRY INFORMATION REQUIRED FOR THE DEVELOPMENT OF A PROCESS FOR THE FLASH HYDROPYROLYSIS OF COAL.

2. PRESENT STUDIES RELATED TO THE RAPID GAS PHASE NON-CATALYTIC HYDROGENATION OF A NON-CAKING COAL (LIGNITE) MAXIMIZING LIQUID HYDROCARBON YIELDS FOR DISTILLATE FUELS AND CHEMICAL FEEDSTOCKS.

PROCESS CHEMISTRY INFORMATION

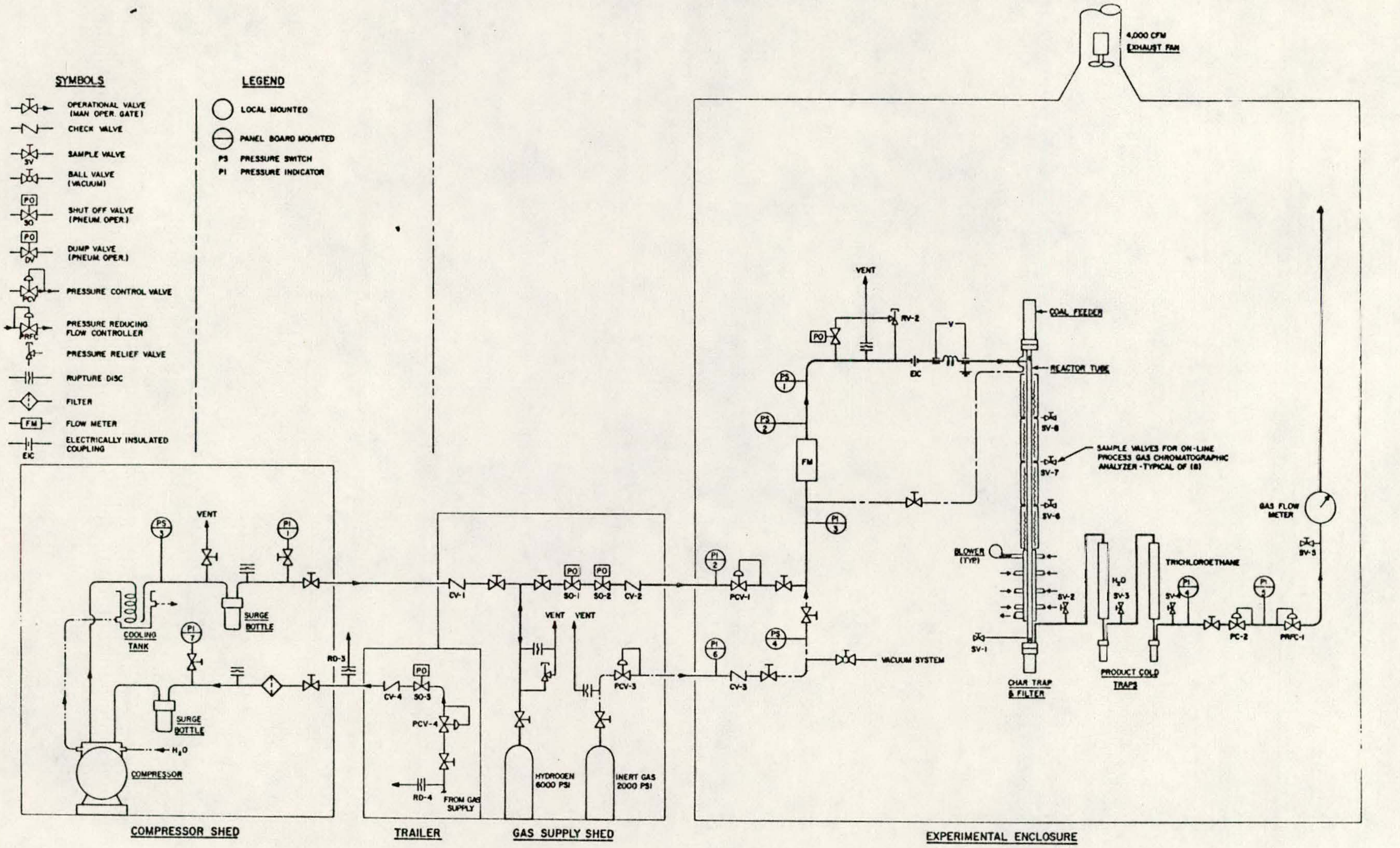
1. CARBON CONVERSION YIELDS AS A FUNCTION OF THE FOLLOWING VARIABLES IN A DOWNFLOW ENTRAINED TUBULAR REACTOR.
   - A. TEMPERATURE
   - B. PRESSURE
   - C. RESIDENCE TIME
     1. COAL
     2. PROCESS GASES
   - D. COAL PREPARATION
     1. GRINDING
     2. DRYING
     3. SIZING
   - E. TYPE OF COAL
     1. LIGNITE NON-CAKING
     2. NON-CAKING SUBBITUMINOUS COAL
   - F. FEED GAS COMPOSITION
   - G. GAS TO COAL FEED RATIO
2. MATERIAL BALANCE CLOSURE ON ALL ELEMENTS, C, H, O, S, N
3. TOTAL PRODUCT CHARACTERIZATION
   - A. CONDENSED LIQUIDS
   - B. GASES
   - C. CHAR
4. DEVELOP PROCESS DESIGN INFORMATION
   - A. KINETIC DATA FOR REACTOR DESIGNS
   - B. MATHEMATICAL MODEL FOR ECONOMIC EVALUATION OF AN INTEGRATED COAL CONVERSION PLANT
   - C. DESIGN INFORMATION FOR A PDU

Accomplishments to Date

1. Completed construction, safety analysis, testing, and placed into operation 1 lb/hr entrained tubular reactor.
   - A. Feed rate in excess of 2 lb/hr
   - B. Temperatures achieved to 815°C
   - C. Operated up to 4000 psi
2. Completed construction and calibration of an on-line process gas chromatograph
   - A. Ten components every 8 minutes
3. Obtained product yield distribution and carbon balance in a series of runs - approximately 100 runs completed
4. Performed baseline and exploratory runs with several different process gases He and CH_4

Design Characteristics of 1" Tubular Reactor Experiment Entrained Down Flow

Reactor size	1 in. I.D. x 12 ft long (8 ft heated)
Material	Inconel 617
Coal flow capacity	up to 2 lb/hr (down to 0.1 lb/hr)
Max. run time	~ 2 hrs (hopper capacity ~ 4 lb)
Hydrogen flow	up to 10 lb/hr (down to 0 flow)
Design pressure	up to 4000 psi
Design temperature	up to 850°C
Coal residence time	~ 0.5 sec (min.) ~ 20 sec (max.) (free fall for 50 μ particles)
Instrumentation	full pressure, temperature, and flow rate remotely controlled
Analytical	On-line gas chromatograph measuring 10 components every 8 min. with integrator and direct readout and compositional analysis. H_2, CO, ($+N_2$), H_2O, CO_2, CH_4, C_2H_6, C_6H_6, C_7H_8, C_8H_{10}, and total HC.
Sample taps	every 2 ft along reactor
Product collection	Trap - char Water condenser - oils Low temp. condenser - BTX Batch gas sampler - gas



COAL FLASH HYDROPYROLYSIS EXPERIMENT

FIG. 1

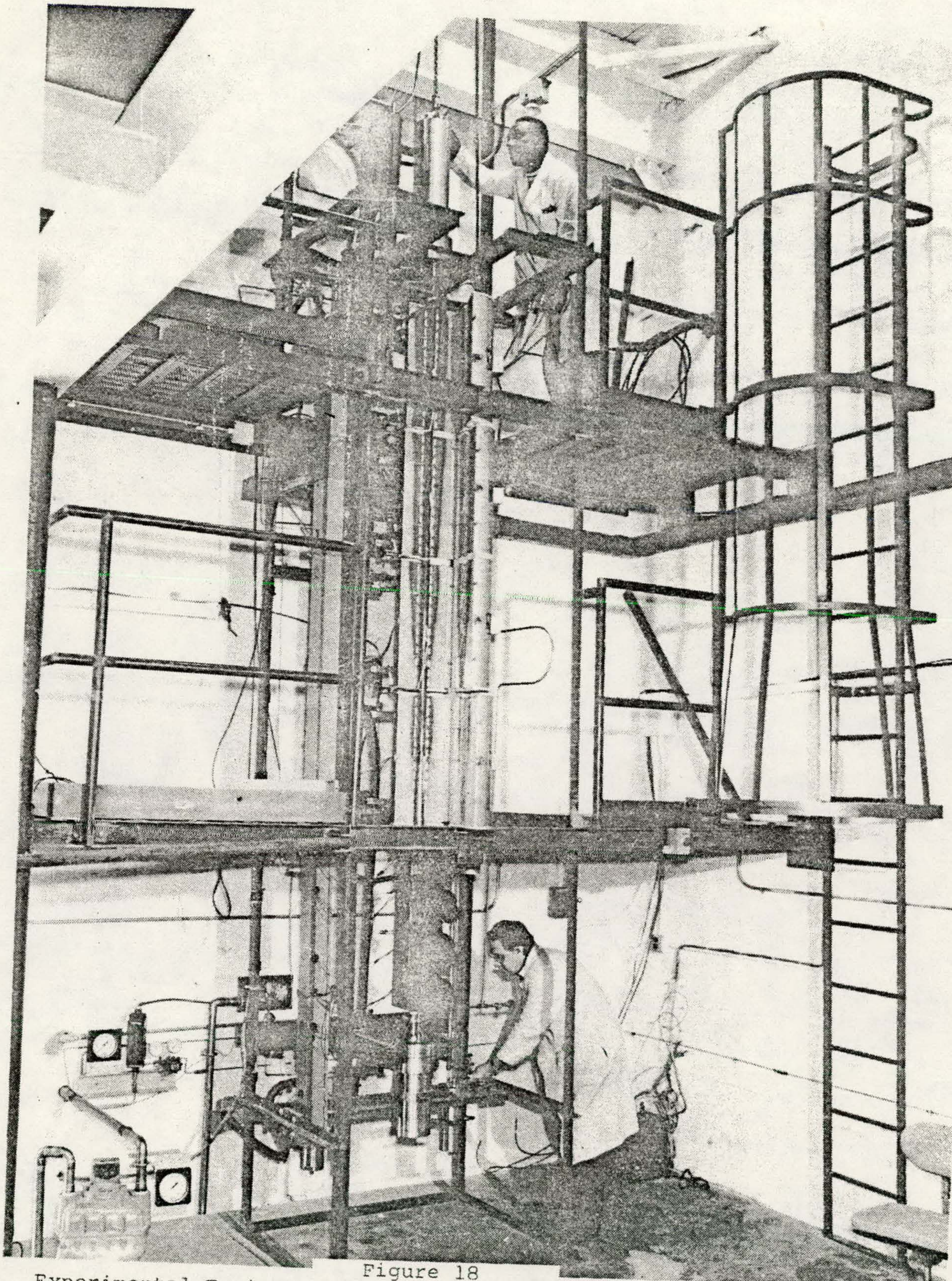


Figure 18
Experimental Equipment Before Addition of Blast Mats and Fireproof Enclosure

RUN NO. 22 SAMPLE NO. 1 DATE 1 -13 - 1977

COAL TYPE- NORTH DAKOTA LIGNITE

COAL RESIDENCE TIME-MEASURED 0. SECONDS
CALCULATED 12.10 SECONDS

NOMINAL CONDITIONS			TOTAL ACCUMULATED WTS.		
COAL FEED RATE	.8199	LB/HR	COAL FED	417.0	GRAMS
HYDROGEN FLOW RATE	1.080	LB/HR	CHAR	180.0	GRAMS
HYDROGEN PRESSURE	2000.	PSI	LIGHT OILS	0.	GRAMS
REACTOR TEMP.	750.0	DEG C	BTX	0.	GRAMS
PREHEAT TEMP.	760.0	DEG C	WATER	0.	GRAMS

	COAL ANALYSIS,%	CHAR ANALYSIS,%
CARBON	59.70	72.22
HYDROGEN	3.800	2.750
OXYGEN	24.39	0.
NITROGEN	.8400	.6900
SULFUR	1.170	1.040
ASH	10.10	23.30

	PRODUCT CONC. IN GAS, LB/C. FT	% CARBON CONV. TO PRODUCT	% MAF COAL CONV. TO PRODUCT
CARBON MONOXIDE	.4149E-03	7.413	11.48
CARBON DIOXIDE	0.	0.	0.
METHANE	.5428E-03	16.93	15.02
ETHYLENE	0.	0.	0.
ETHANE	.2415E-03	8.039	6.682
BENZENE	.2166E-03	8.326	5.993
TOLUENE	0.	0.	0.
XYLENE	0.	0.	0.
LIGHT OIL	.1757E-03	6.758	4.861
WATER	.3831E-03	0.	10.60
TOTAL		47.47	54.63

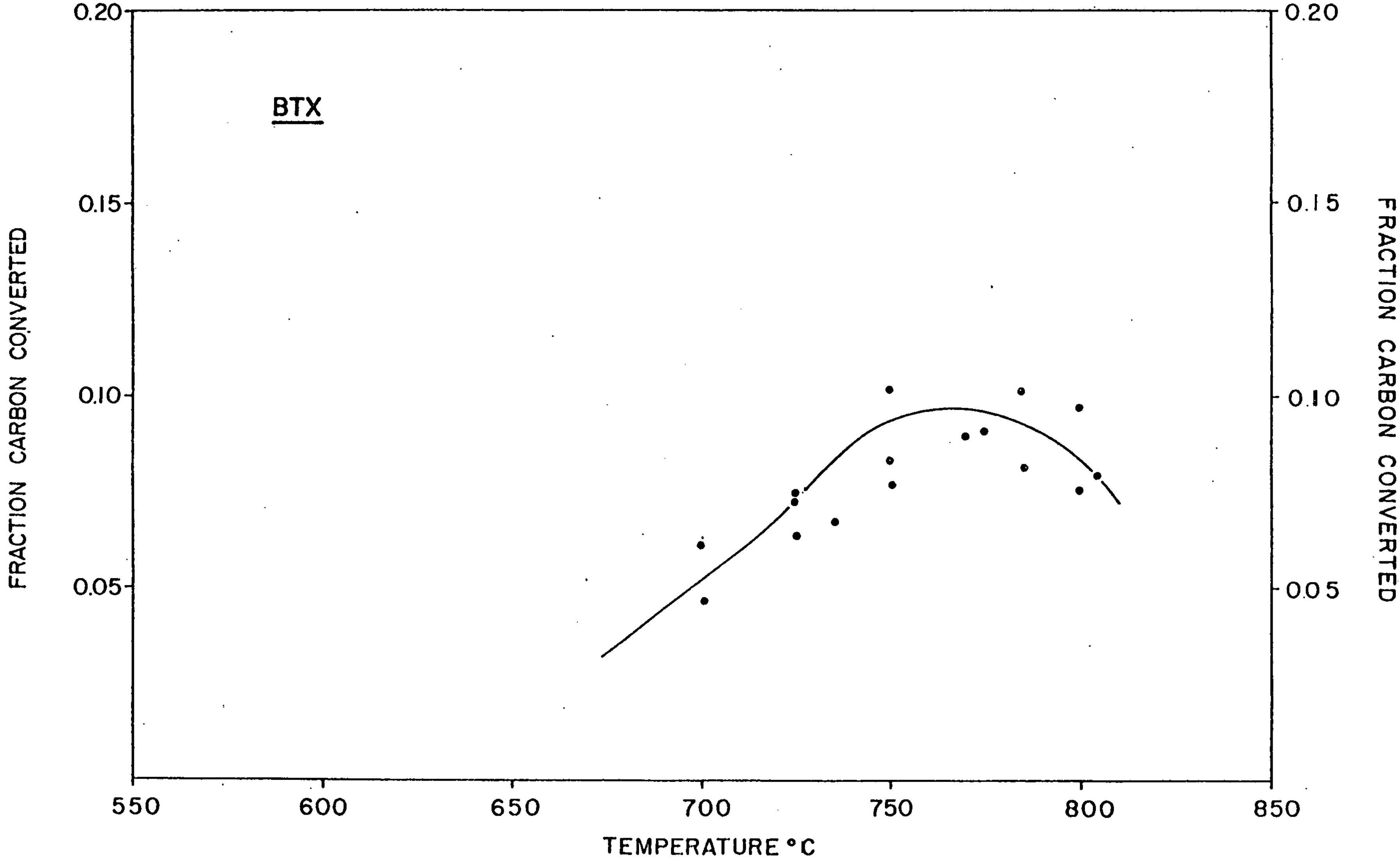
TOTAL MATERIAL BALANCE, %

	CARBON	HYDROGEN	OXYGEN	NITROGEN	SULFUR	MAF COAL
LIQUIDS	15.1	19.8	0.	0.	0.	10.9
HYDROCARBON GASES	25.0	121.	0.	0.	0.	21.7
CARBON OXIDES	7.41	0.	24.2	0.	0.	11.5
WATER	0.	27.9	34.7	0.	0.	10.6
AMMONIA	0.	3.06	0.	64.5	0.	.732
HYDROGEN SULFIDE	0.	1.19	0.	0.	61.6	.852
CHAR	52.2	31.2	0.	35.5	38.4	36.8
TOTAL	99.7	204.	58.9	100.	100.	93.0

HYDROGEN CONSUMPTION- .3961E-01 LB HYDROGEN/LB COAL
.7962 MOLE HYDROGEN/MOLE CARBON

Table VI

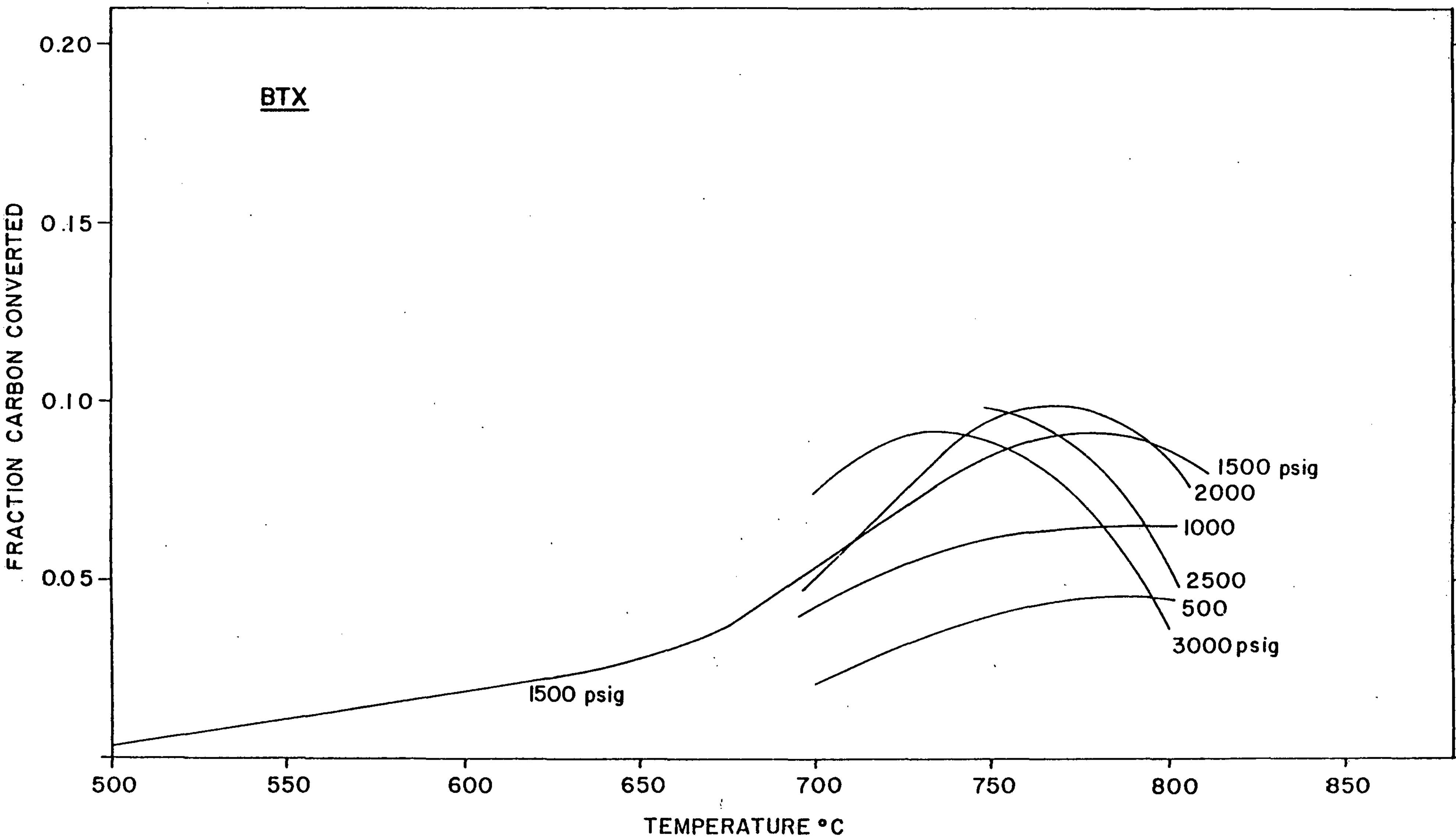
Computer Data Reduction and Material Balance Printout



FLASH HYDROPYROLYSIS OF LIGNITE

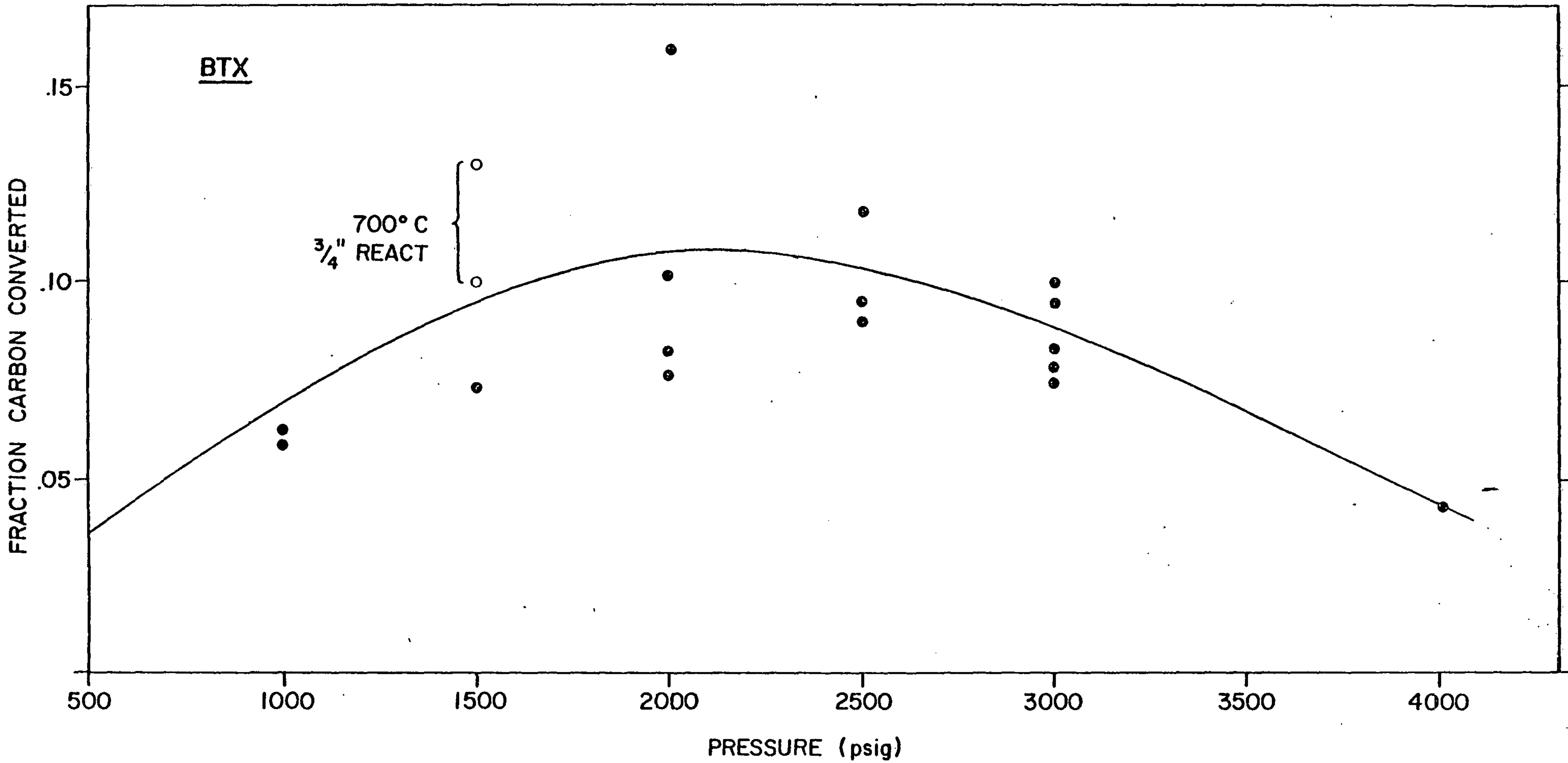
BTX vs TEMPERATURE

PRESS. 2000 psig

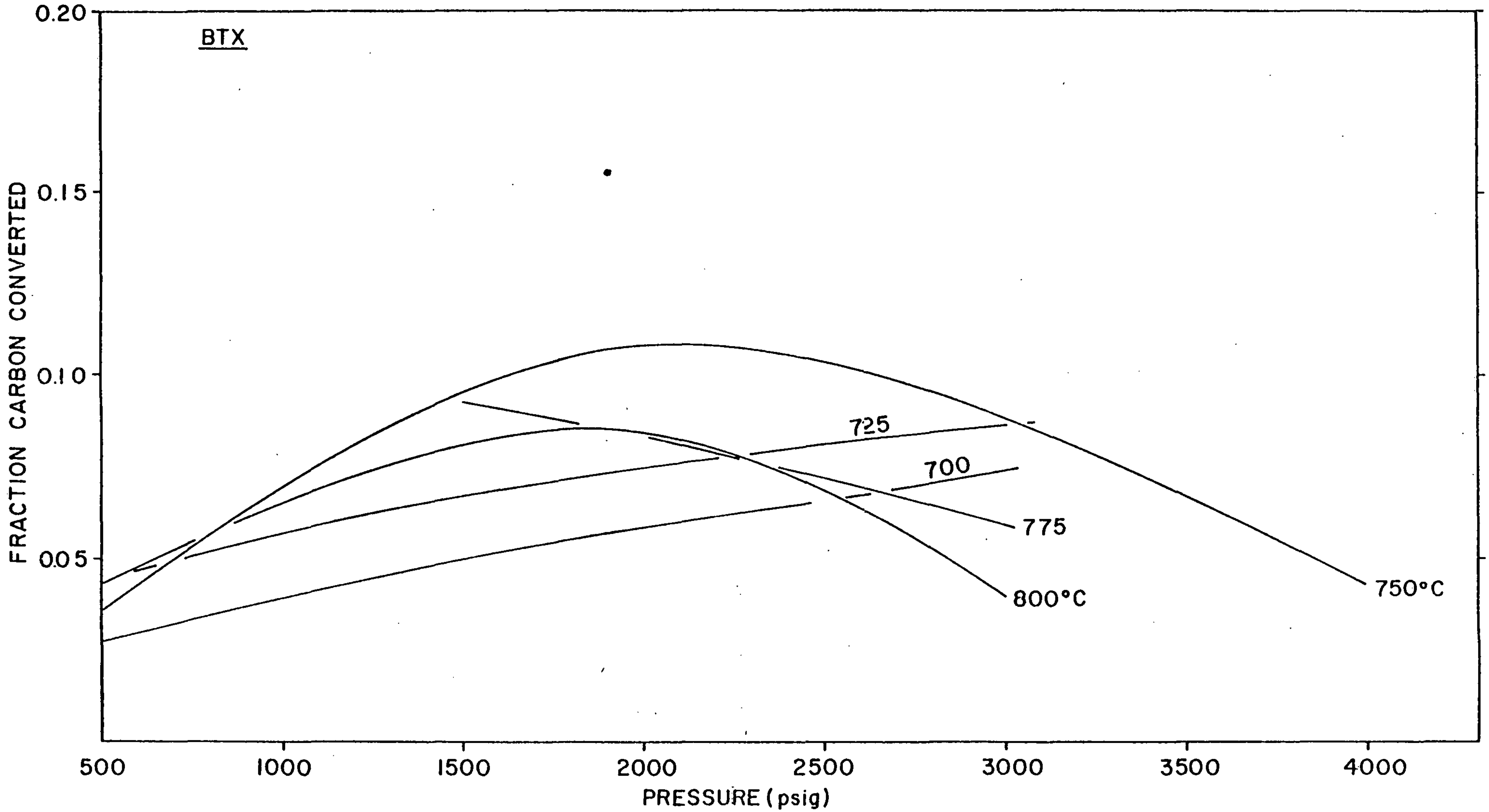


FLASH HYDROPYROLYSIS OF LIGNITE

BTX vs TEMPERATURE



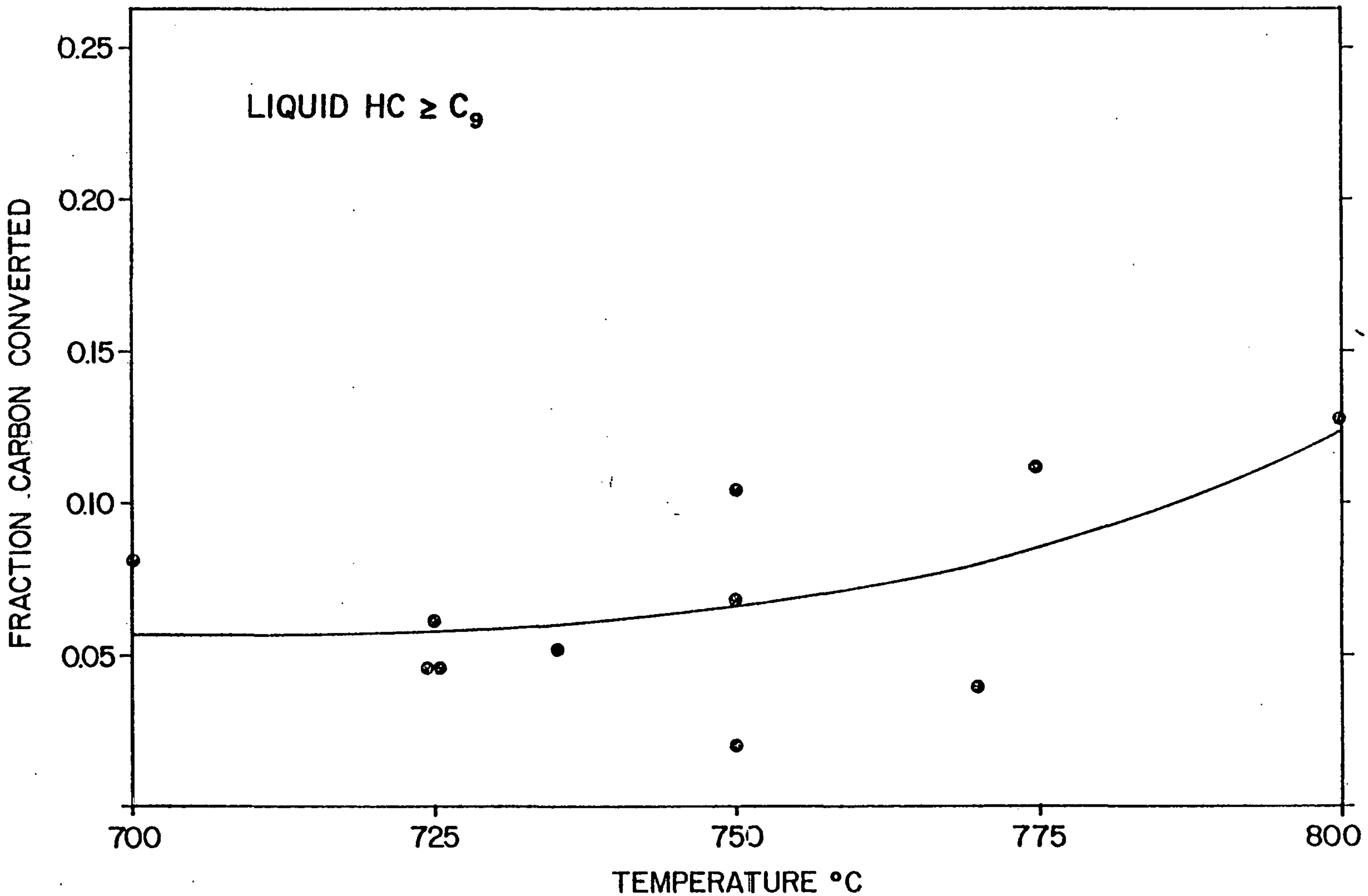
FLASH HYDROPYROLYSIS OF LIGNITE
BTX vs. PRESSURE
TEMP. 750°C



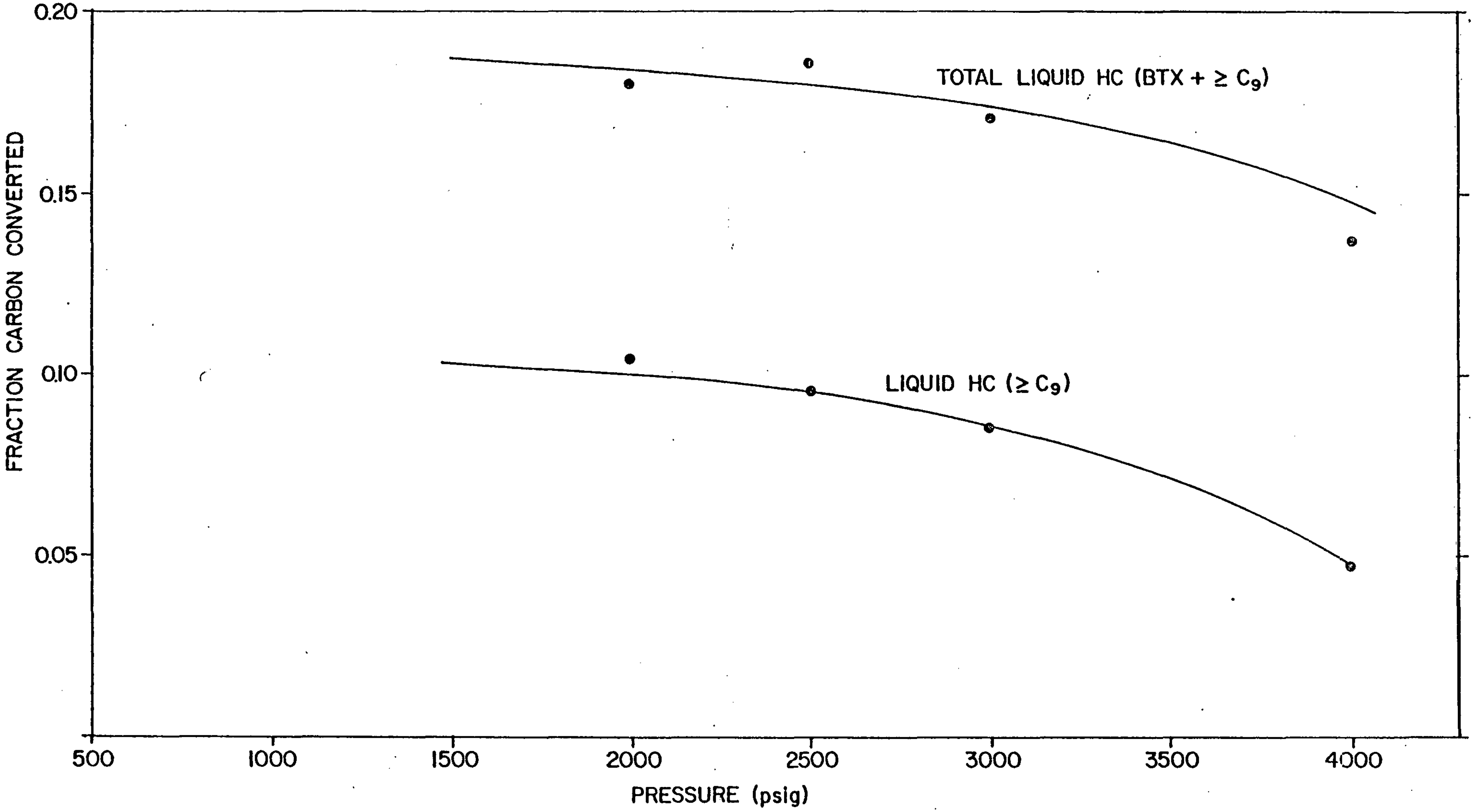
FLASH HYDROPYROLYSIS OF LIGNITE

BTX vs PRESSURE

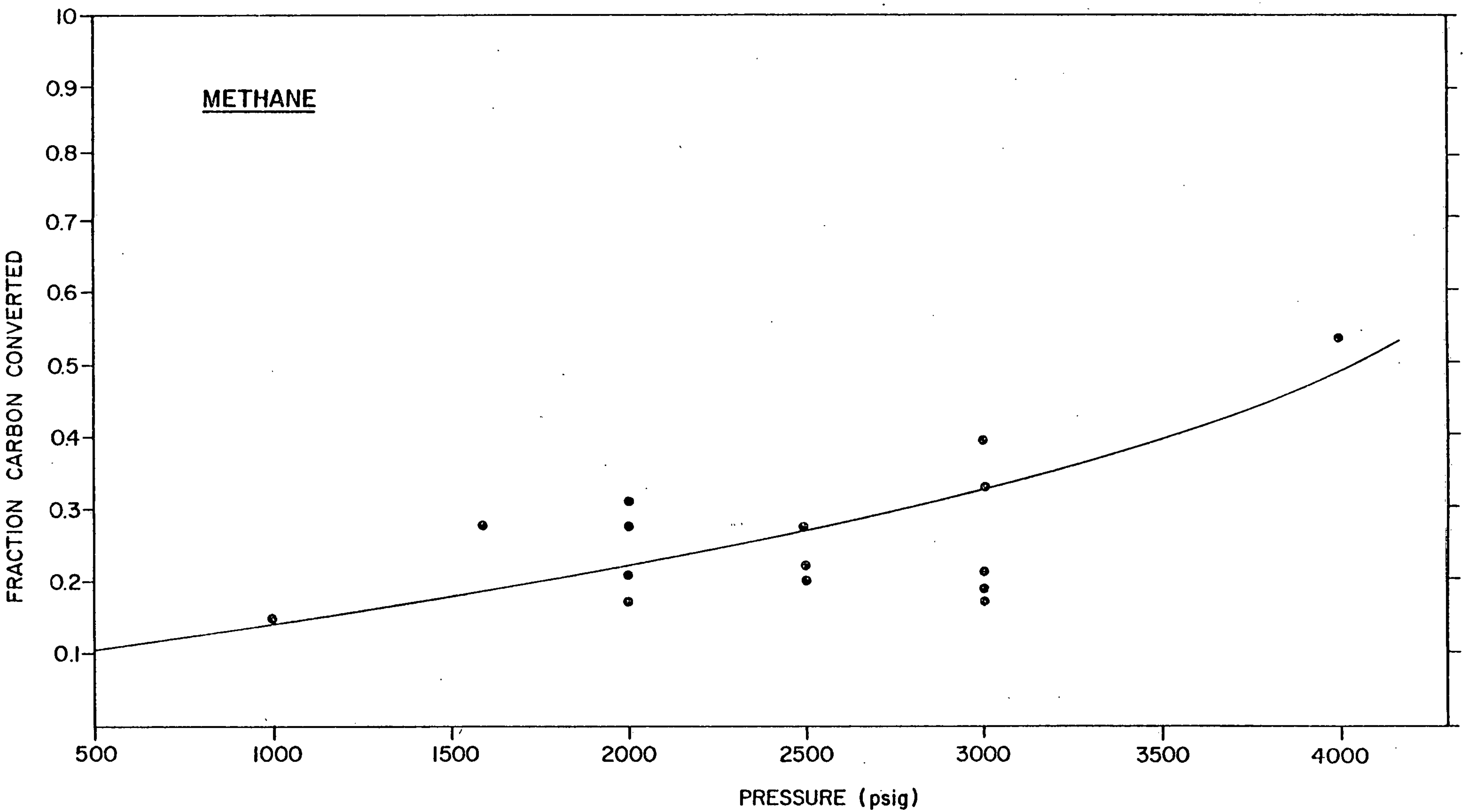
TEMP. 700 - 800°C



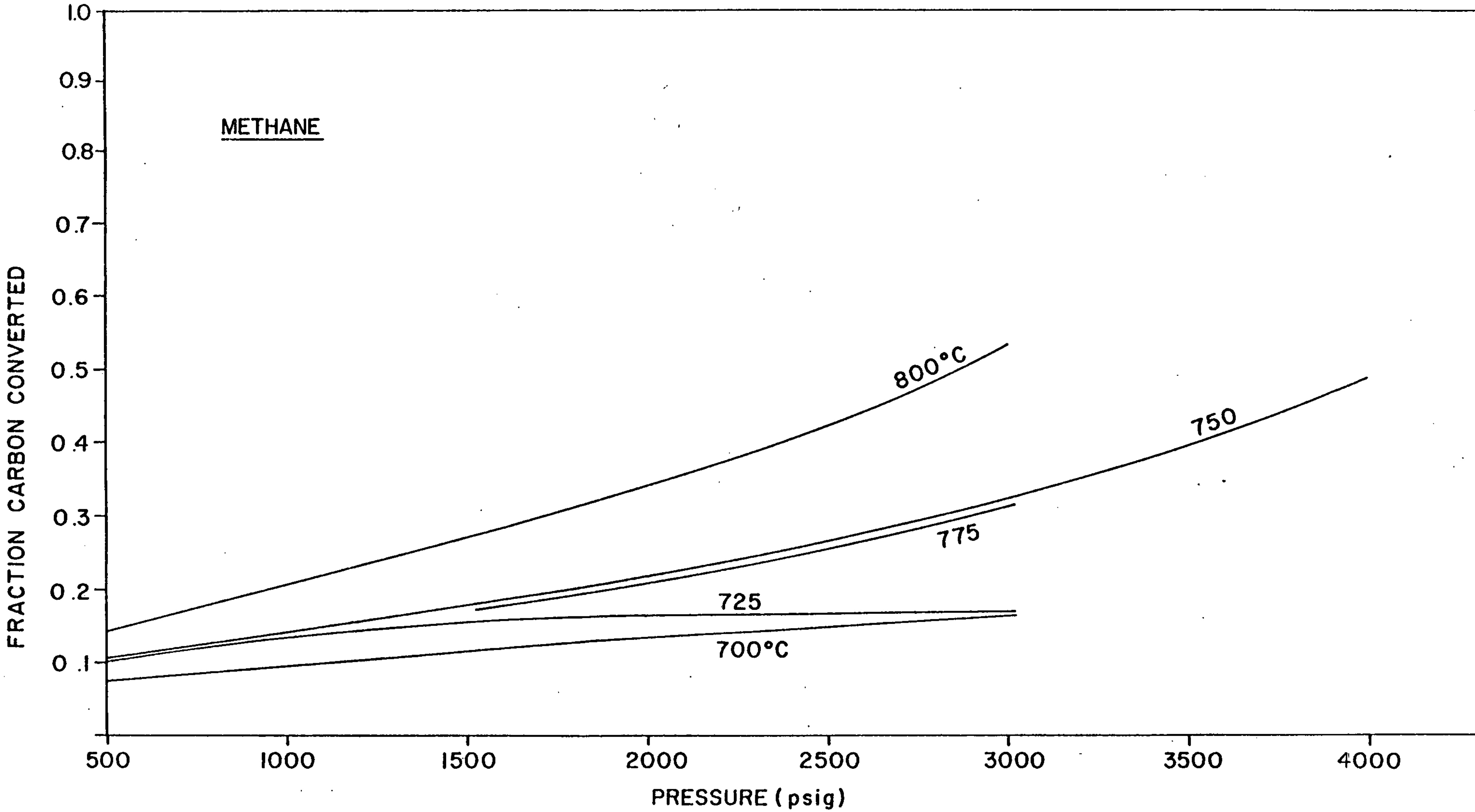
FLASH HYDROPYROLYSIS OF LIGNITE
LIQUID HC ≥ C_9 vs. TEMPERATURE
AT 2000 psig



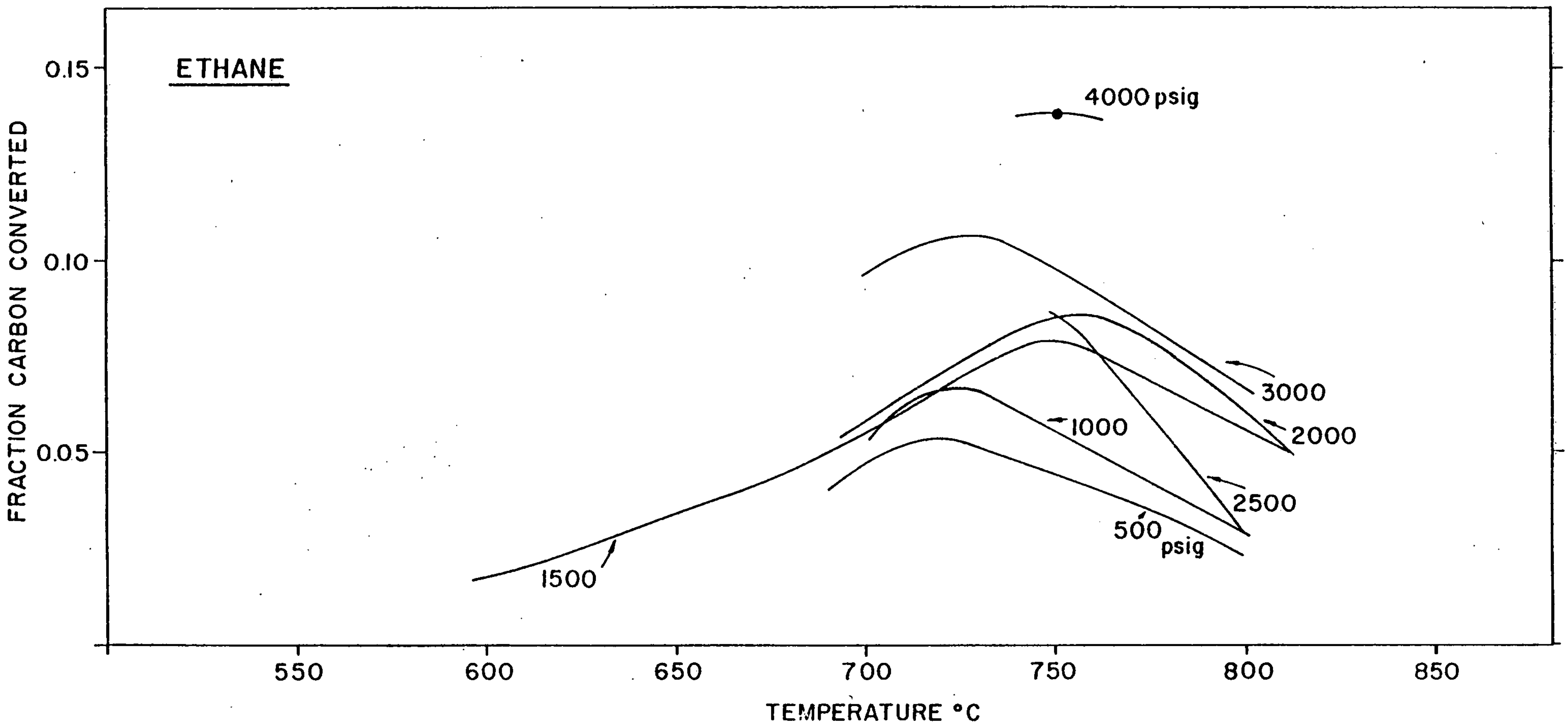
FLASH HYDROPYROLYSIS OF LIGNITE
TOTAL LIQUID AND LIQUID HC (≥ C_9) YIELDS vs. PRESSURE
AT 750°C



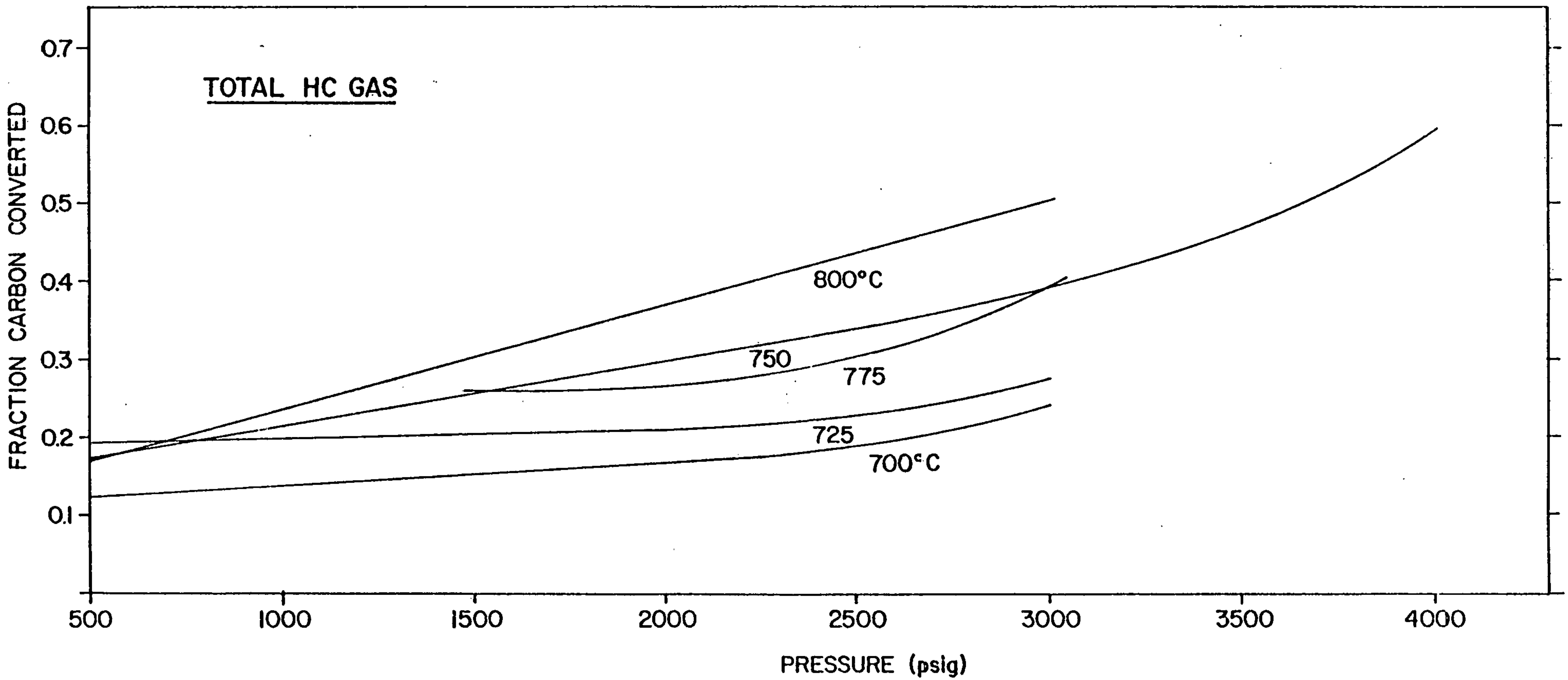
FLASH HYDROPYROLYSIS OF LIGNITE
METHANE vs. PRESSURE
TEMP. 750° C



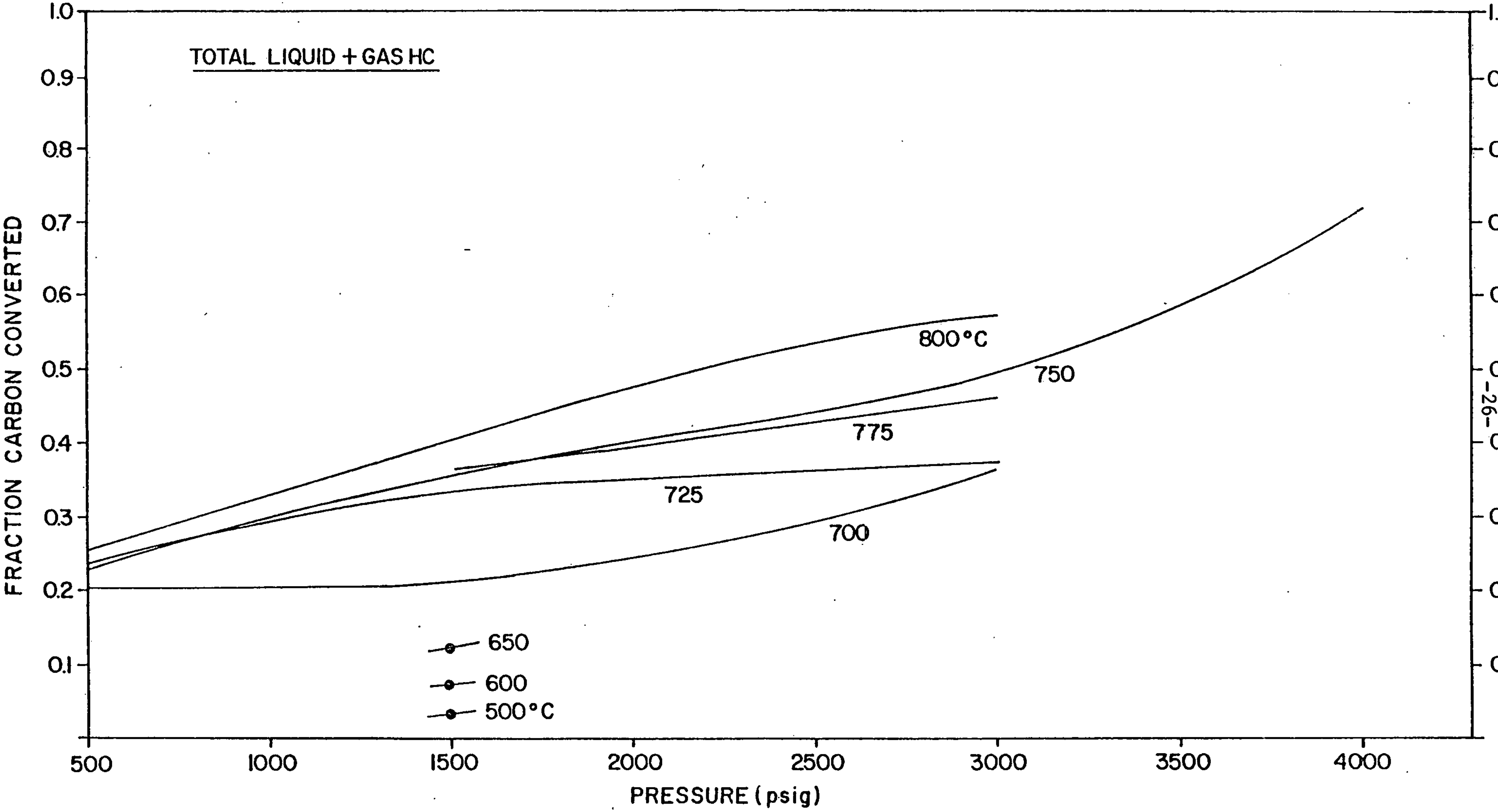
FLASH HYDROPYROLYSIS OF LIGNITE
METHANE vs PRESSURE
TEMP. 700-800°C



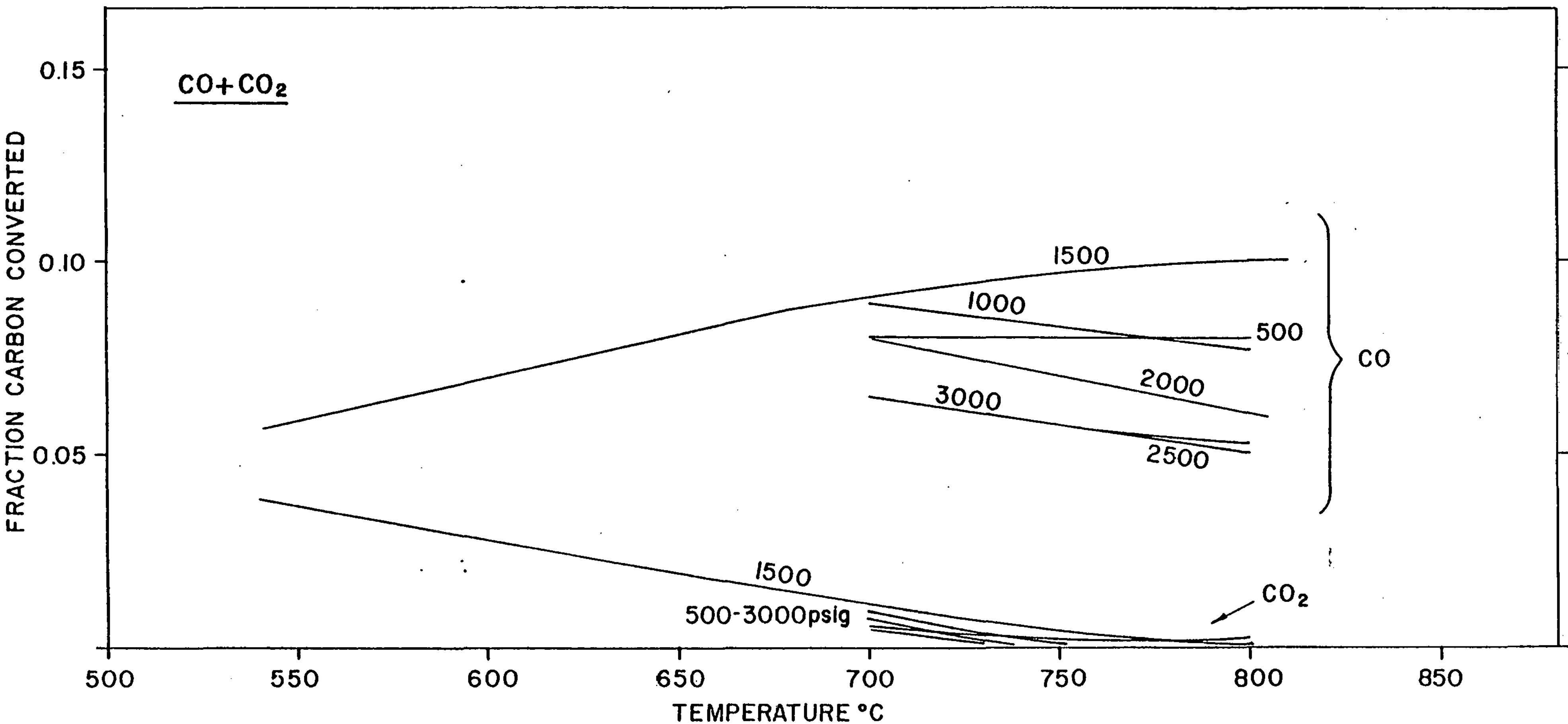
FLASH HYDROPYROLYSIS OF LIGNITE
ETHANE vs TEMPERATURE
PRESS. 500 - 4000 psig



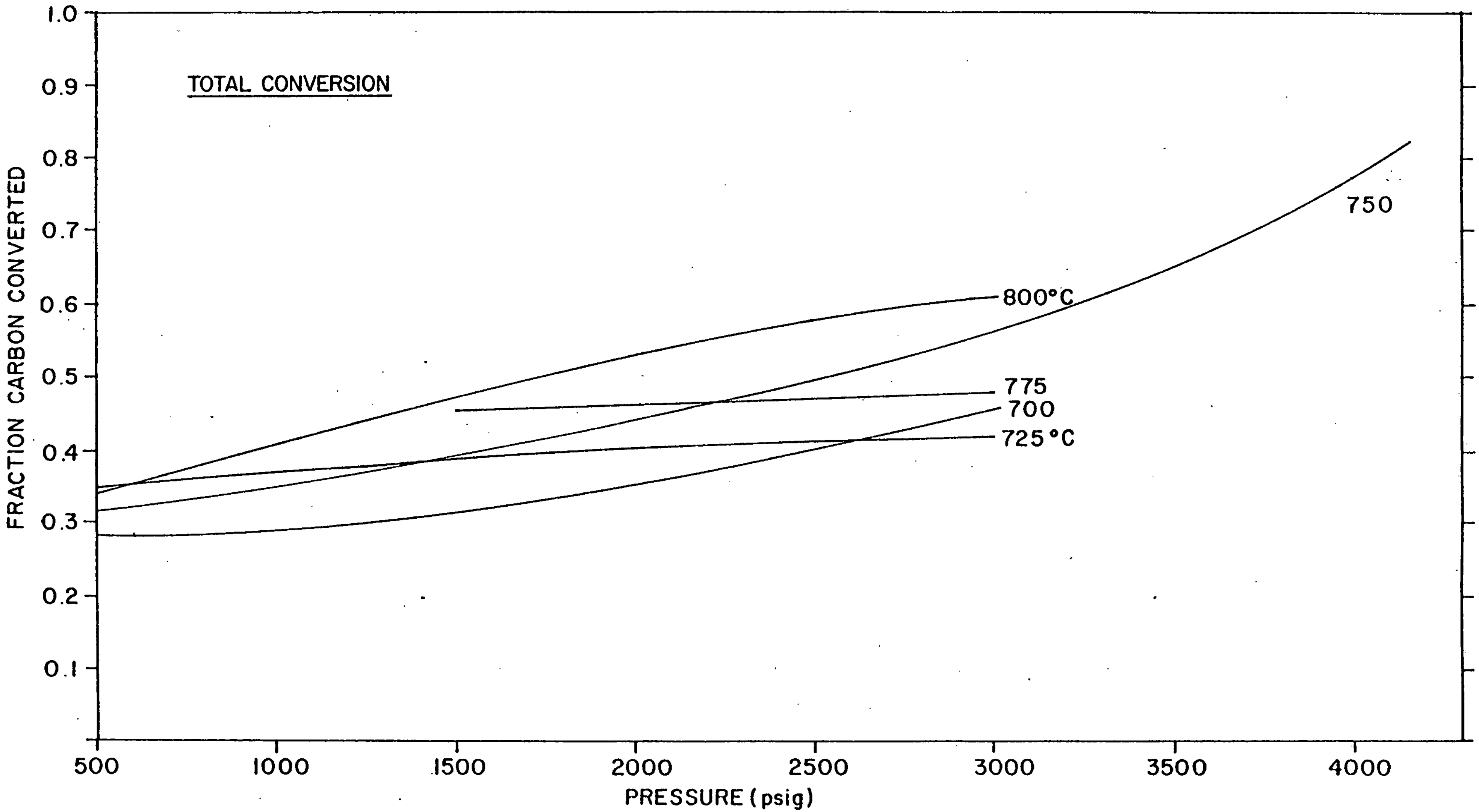
FLASH HYDROPYROLYSIS OF LIGNITE
TOTAL HC GAS vs. PRESSURE
TEMP. 700°- 800°C



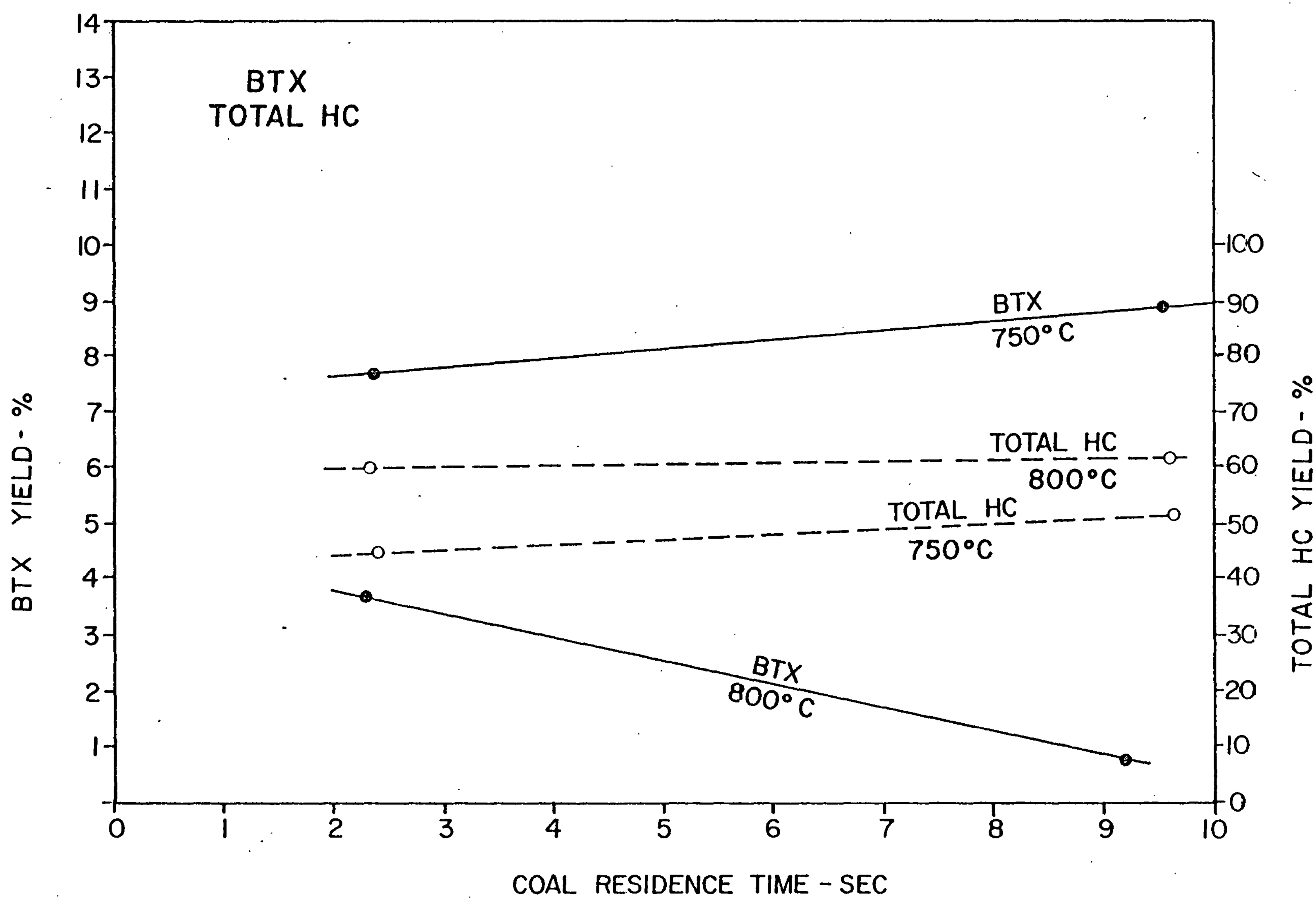
FLASH HYDROPYROLYSIS OF LIGNITE
TOTAL LIQUID +GAS HC vs PRESSURE
TEMP. 500 - 800°C



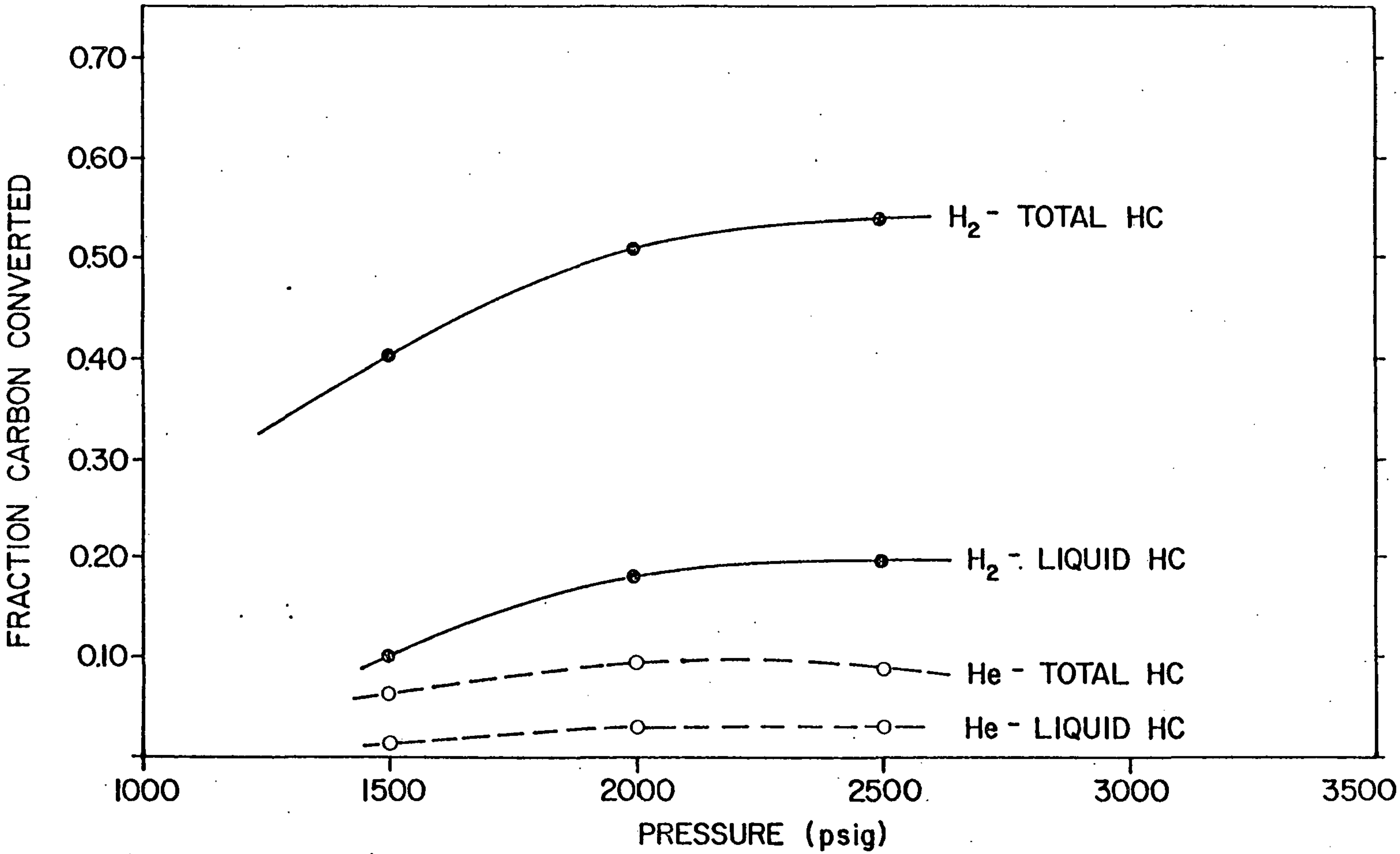
FLASH HYDROPYROLYSIS OF LIGNITE
$CO+CO_2$ vs TEMPERATURE
PRESS. 500 - 3000 psig



FLASH HYDROPYROLYSIS OF LIGNITE
TOTAL CARBON CONVERSION TO GASES AND LIQUIDS vs PRESSURE
TEMP. 700-800°C



FLASH HYDROPYROLYSIS OF LIGNITE
BTX AND TOTAL HC vs. RESIDENCE TIME
2500 psig



FLASH HYDROPYROLYSIS OF LIGNITE
TOTAL HC AND LIQUID YIELD vs. PRESSURE
TEMP. = 750°C

FLASH HYDROPYROLYSIS OF LIGNITE

AVERAGE CONVERSIONS (% OF C FEED)

TEMP. - 750°C

PRESSURE, PSIG	1500	2000	2500
BTX (BENZENE)	9.5	10.8	10.5
HEAVY HC $\geq C_9$	8.8	10.4	9.5
METHANE	18.0	22.0	27.0
ETHANE	7.9	8.5	8.5
CO +(CO_2)	10.0	7.0	6.0
TOTAL HC GAS	25.9	30.5	35.5
TOTAL HC LIQUID	18.3	21.1	20.0
TOTAL HC CONVERSION	44.2	51.7	55.5
TOTAL CONVERSION	54.2	58.7	61.5

FLASH HYDROPYROLYSIS OF LIGNITE

RUN NO.	73
PRESSURE, PSIG	4000
TEMPERATURE ^0C	750^0

CONVERSION	% OF C
BTX (BENZENE)	4.3
HEAVY HC $\geq C_9$	5.7
METHANE	52.8
ETHANE	13.9
CO	4.5
CO_2	0.2
TOTAL	81.4

Table V

Composition of Polynuclear Aromatic

Hydrocarbons (PAHs) in Flash Hydropyrolysis Product

	Tentatively Identified PAH	% by wt
1	Naphthalene	47.63
2	2-Methyl Naphthalene	5.57
3	1-Methyl Naphthalene + Azulene	2.12
4	Biphenyl	4.97
5	2,6-Dimethyl Naphthalene	0.16
6	1,3 and/or 1,6-Dimethyl Naphthalene	0.42
7	1,5 and/or 2,3-Dimethyl Naphthalene	0.15
8	1,2-Dimethyl Naphthalene and/or Acenaphthylene	0.14
9	Acenaphthene	1.10
10	Fluorene	8.39
11	9,10-Dihydroanthracene	0.52
12	9-Methyl Fluorene	0.74
13	1-Methyl Fluorene	0.50
14	Phenathrene	12.89
15	Anthracene	0.97
16	2-Methyl Anthracene	0.14
17	1-Methyl Phenathrene	0.37
18	Fluoranthene	1.98
19	Pyrene	4.41
20	1,2-Benzofluorene	0.91
21	2,3-Benzofluorene	1.02
22	1-Methyl Pyrene	0.08
23	1,2-Benzanthracene	0.53
24	Chrysene	1.01
25	7,12-Dimethyl-1,2,Benzanthracene	0.15
26	1,2-Benzopyrene[1] (BaP)	0.98
27	Perylene	0.36
28	3-Methyl Cholanthrene	0.15
29	O-phenylene pyrene	0.55
30	Picene	0.04
31	1,12-Benzoperylene	0.78
32	Anthanthrene	0.27
		100.00

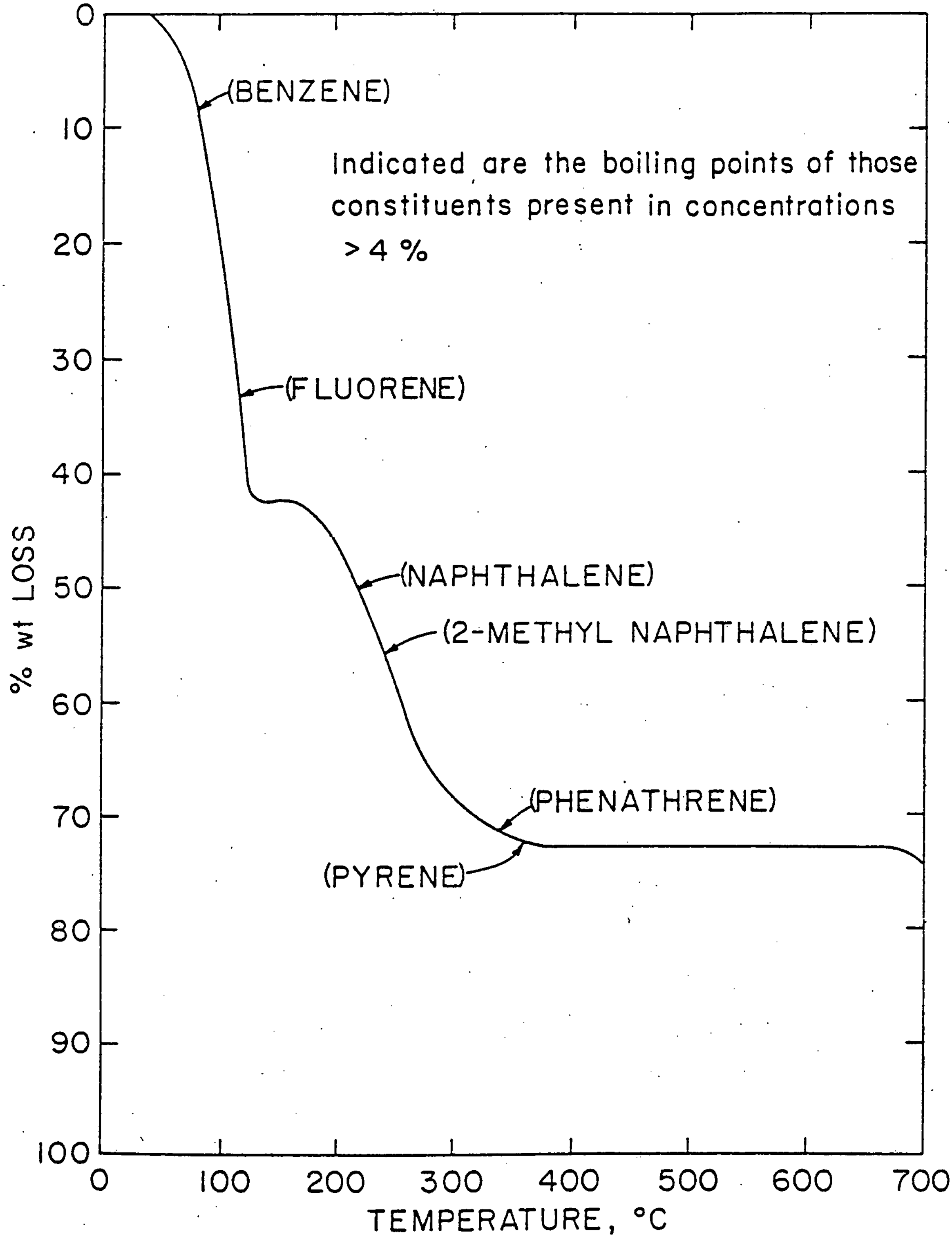
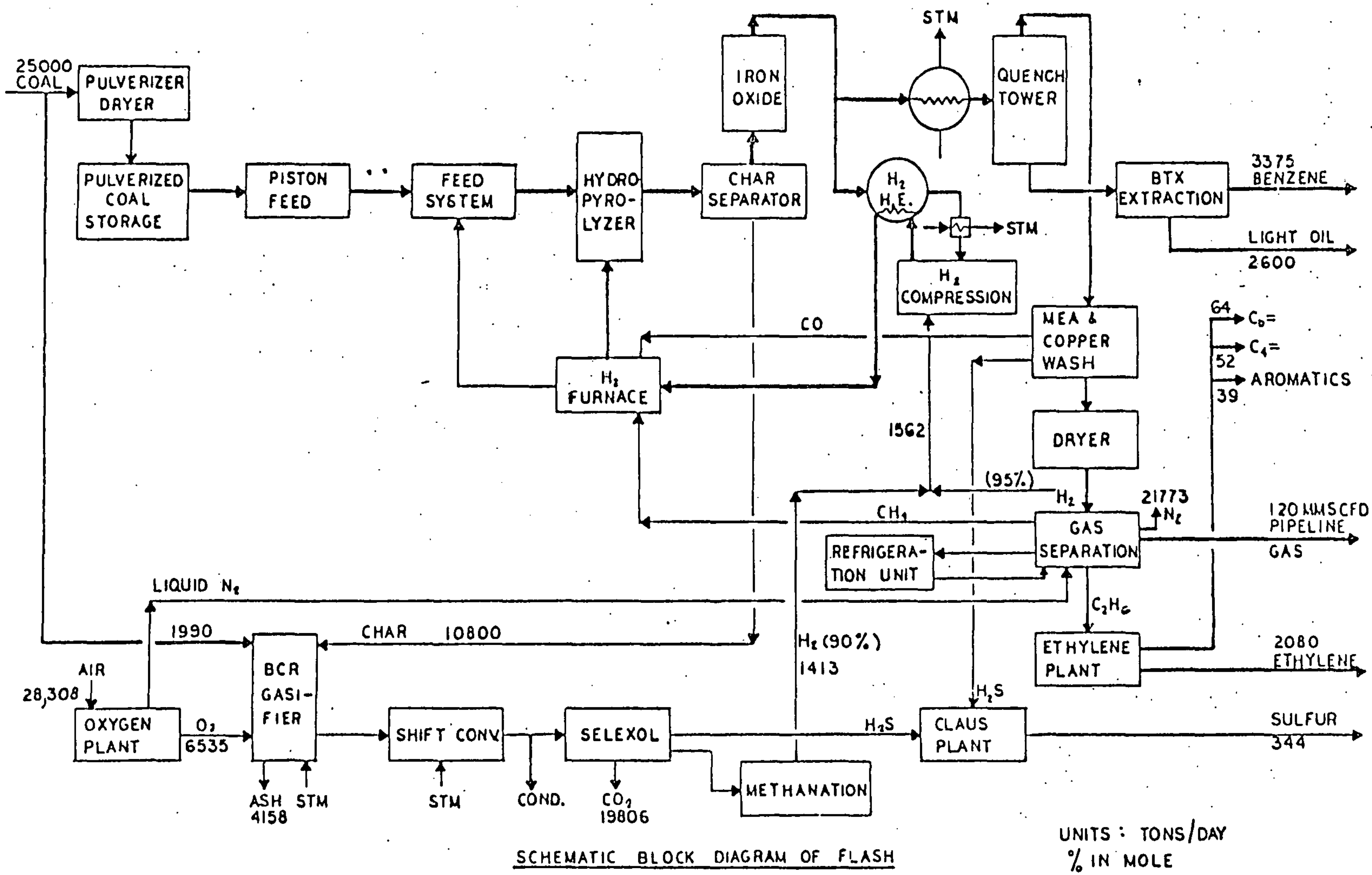


Figure 1: FLASH HYDROPYROLYSIS OF LIGNITE
THERMOGRAVIMETRIC ANALYSIS (TGA) OF 32 mg LIQUID SAMPLE

FLASH HYDROPYROLYSIS OF COAL
FUTURE WORK

1. COMPLETE PARAMETRIC PROCESS CHEMISTRY RUNS FOR LIGNITE
   A) COMPLETE PRESSURE SCAN 500 PSI - 4000 PSI
   B) COMPLETE TEMPERATURE SCAN 500°C - 850°C
2. RESIDENCE TIME SCAN
   A) TAKE ANALYSIS ALONG REACTOR LENGTH
   B) VARY COAL AND HYDROGEN FLOW
3. RECYCLE CHAR AND ASH
   A) ADD TO COAL FEED
4. INVESTIGATE FEED GAS COMPOSITION
   A) METHANE AND ETHANE
   B) CO AND CO_2
   C) H_2O
5. COAL PREPARATION
   A) EFFECT OF PARTICLE SIZE
   B) EFFECT OF MOISTURE
6. DETERMINE EFFECT OF TYPE OF COAL
   A) SUBBITUMINOUS
   B) BITUMINOUS
7. UTILIZE CATALYTIC AGENTS
   A) HETEROGENEOUS
   B) HOMOGENEOUS
8. CONVERT UNIT TO LONGER CONTINUOUS OPERATION
   A) LOCK HOPPERS
   B) GAS RECIRCULATION
9. COLD FLOW EXPERIMENTS FOR REACTOR DESIGN ANALYSIS



SCHEMATIC BLOCK DIAGRAM OF FLASH HYDROPYROLYSIS OF COAL TO CHEMICAL FEEDSTOCKS AND PIPELINE GAS

FLASH HYDROPYROLYSIS OF COAL
EVALUATION

COAL ANALYSIS

CARBON	60%
HYDROGEN	4
OXYGEN	24
NITROGEN	I
SULFUR	I
ASH	I0
	I00

ASSUMED CARBON DISTRIBUTION IN PRODUCTS

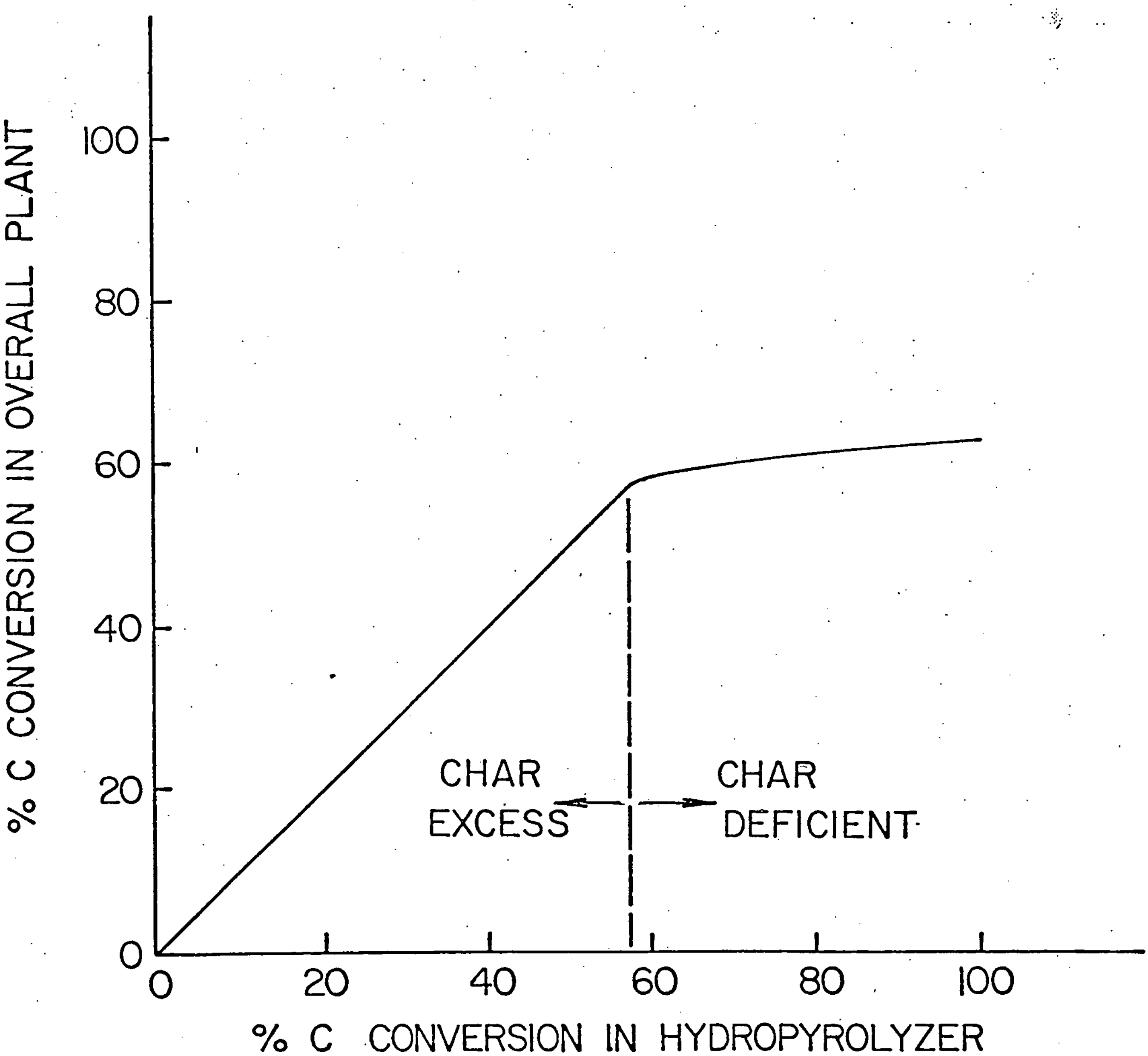
METHANE	37.5
ETHANE	I5.5
BTX	I6.3
LIGHT OIL	I3.2
CARBON OXIDES	I7.5
	I00.0

COAL FEED TO REACTOR 25,000 T/D

ALL OXYGEN CONSUMED TO PRODUCE CO_X AND H_2O

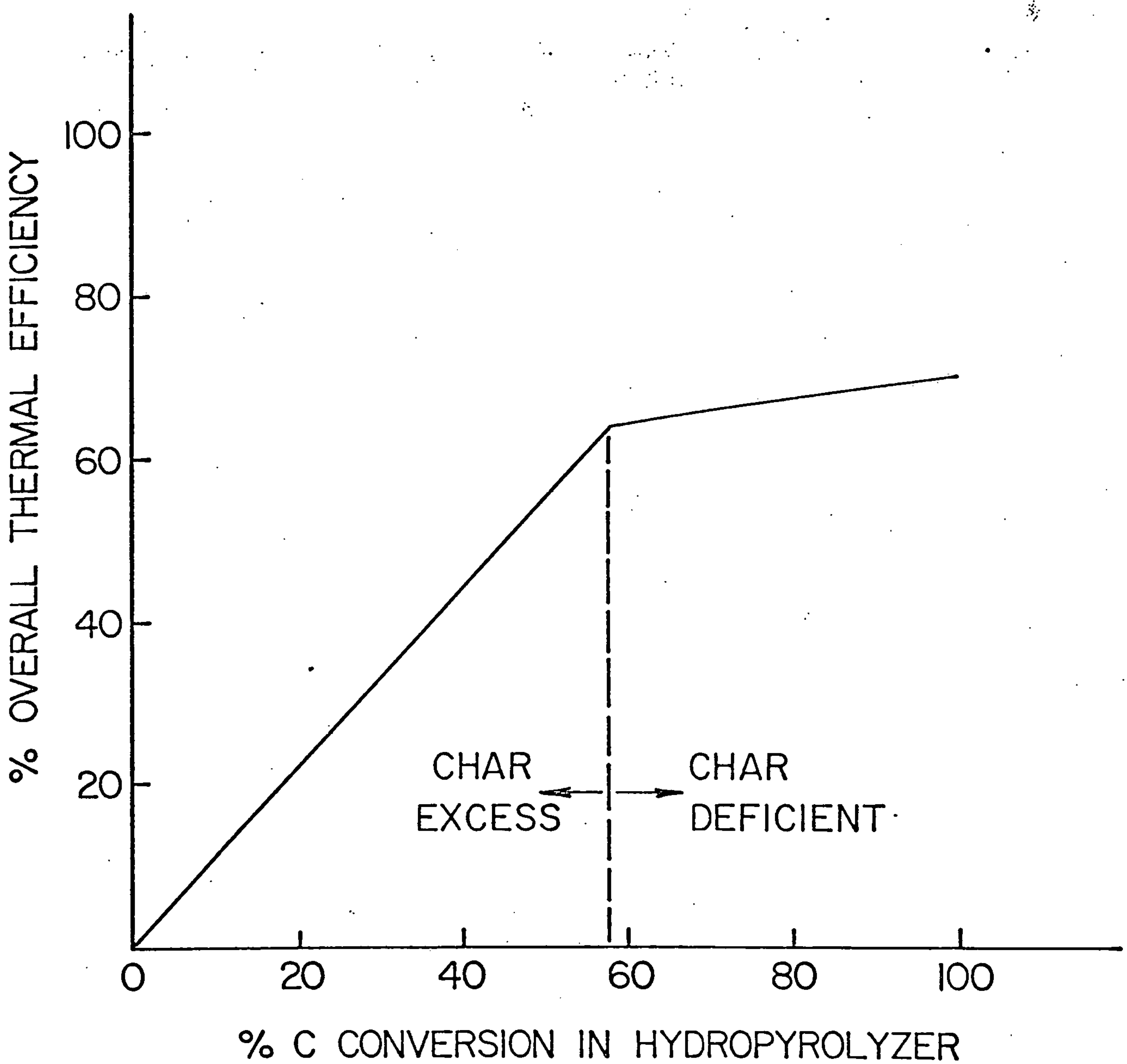
FLASH HYDROPYROLYSIS OF COAL

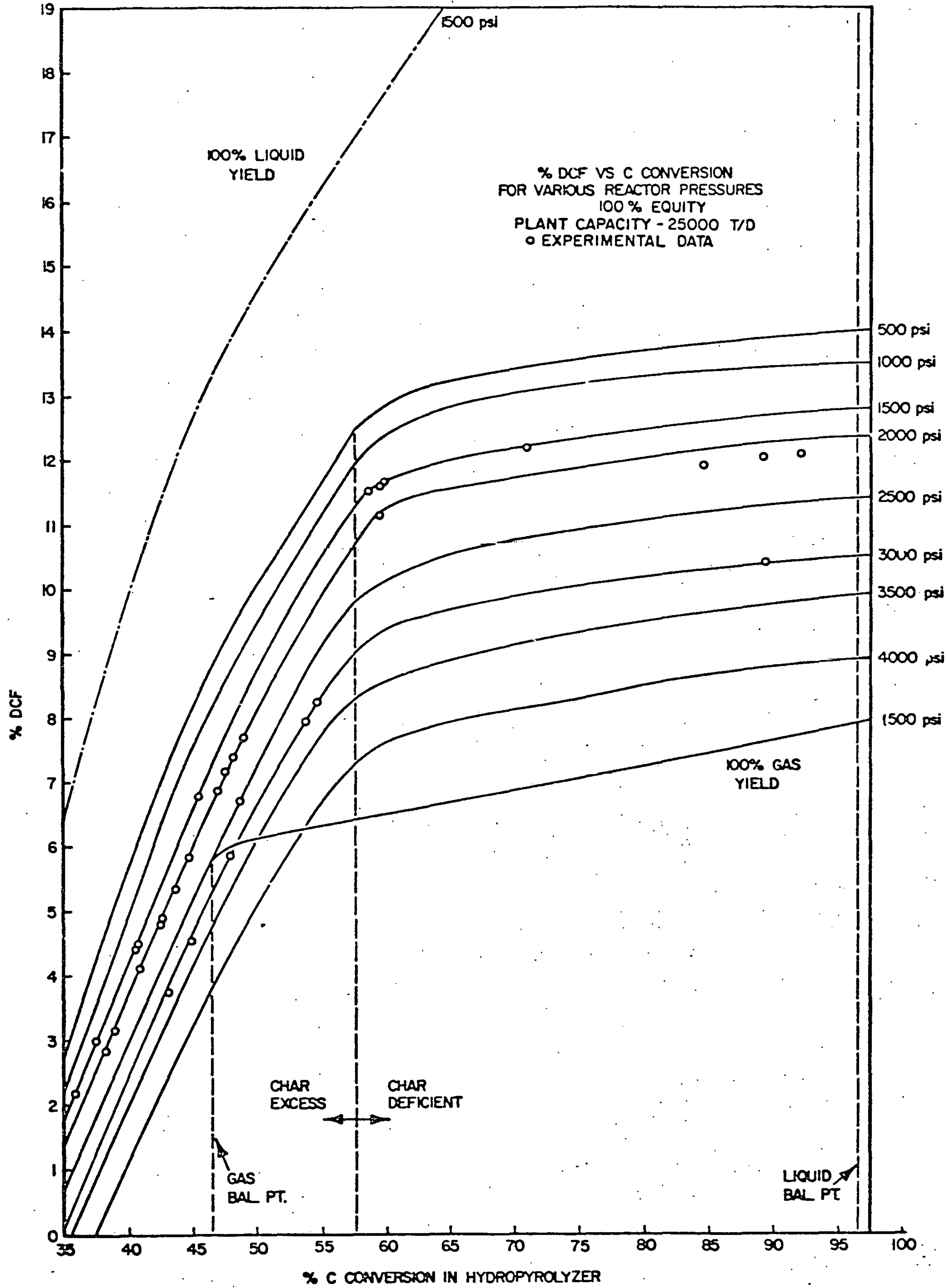
OVERALL PLANT C CONVERSION VS C CONVERSION IN HYDROPYROLYZER



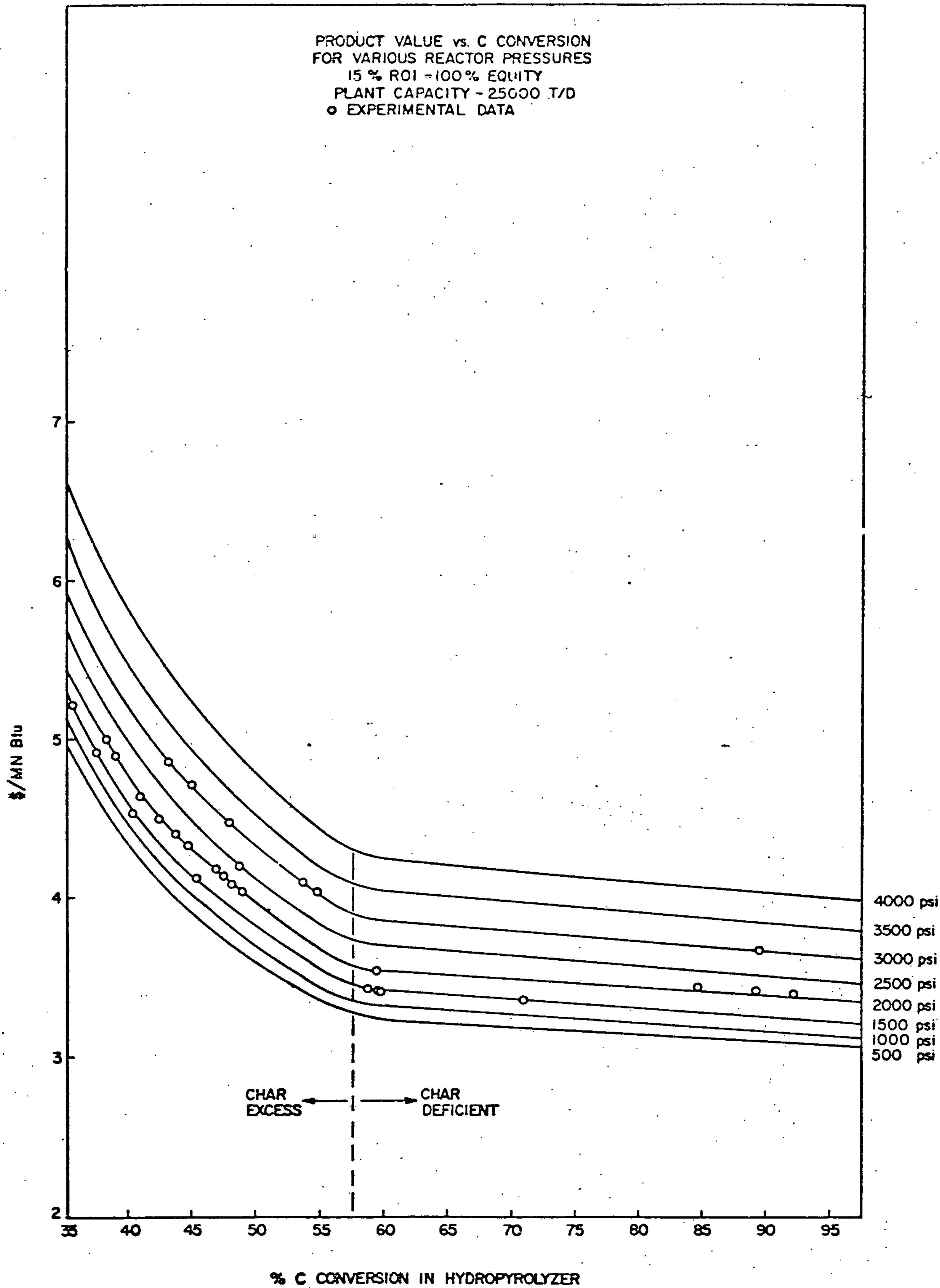
FLASH HYDROPYROLYSIS OF COAL

OVERALL THERMAL EFFICIENCY VS C CONVERSION IN HYDROPYROLYZEF





FLASH HYDROPYROLYSIS OF LIGNITE



FLASH HYDROPYROLYSIS OF LIGNITE