



DE82004148

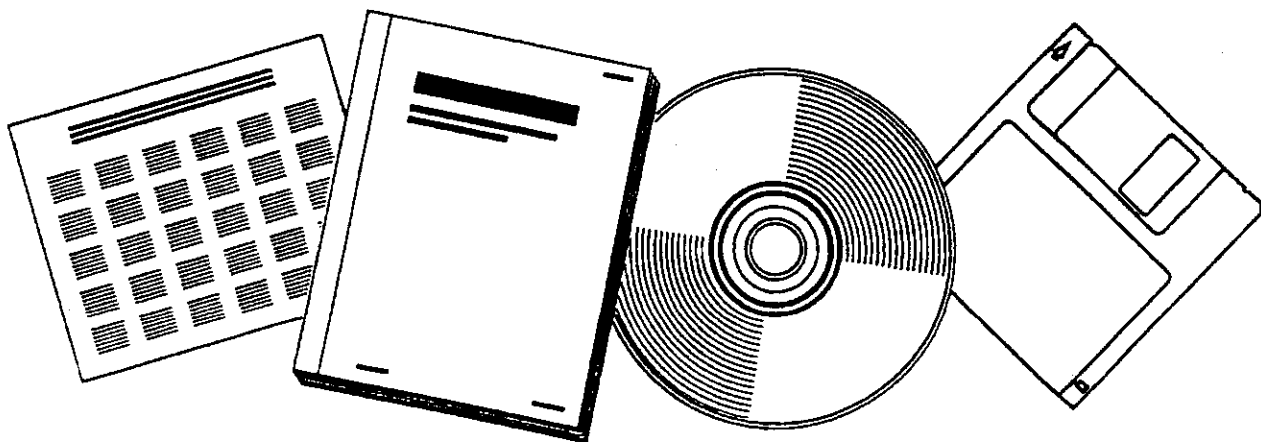
40776-2

NTIS[®]
Information is our business.

**FISCHER-TROPSCH SYNTHESIS IN
SLURRY-REACTOR SYSTEMS. QUARTERLY REPORT,
AUGUST 1, 1981-OCTOBER 31, 1981**

MASSACHUSETTS INST. OF TECH., CAMBRIDGE

1981



U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

DOE/PC/40771--2

Fischer-Tropsch Synthesis in Slurry-Reactor Systems

MASTER

Quarterly Report for Period

August 1, 1981 to October 31, 1981

DOE/PC/40771--2

DE82 004148

Department of Chemical Engineering

and

Energy Laboratory

Massachusetts Institute of Technology

Cambridge, Massachusetts

Report No.: DOE/PC40771-2

Grant No.: DE-FG22-81PC40771

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof nor any of their employees makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process or service by trade name, trademark, manufacturer or otherwise does not necessarily constitute or imply its endorsement, recommendation or approval by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Submitted by

C.N. Satterfield

T. Bartos

G.A. Huff, Jr.

E. Stenger

I. SUMMARY

A large quantity of data were obtained with the fused iron catalyst under intrinsic kinetic conditions, covering for the first time 50 and 200 psi. These data are being analyzed for information about overall rates and product selectivity. Preliminary conclusions about a rate model are presented.

Study of the effects of suspended solids on gas-liquid mass transfer was started. Most previous information is on aqueous systems, which is not readily translatable into predicted effects in organic liquids such as Fischer-Tropsch liquids. The most promising and useful method at present appears to be a chemical method based on absorption of CO_2 into an organic solution of an amine. We are currently studying its potential.

A report has been prepared describing the detailed design of our reactor and trapping procedures, as follows: George A. Huff, Jr. and Charles N. Satterfield, "A Stirred Autoclave Apparatus for Study of the Fischer-Tropsch Synthesis in a Slurry-Bed I. Reactor and Trapping Procedures." A copy is appended.

II. DETAILED RESULTS

A. Reaction Studies

In accordance with the projected plans in our last report, an expanded base of intrinsic kinetic data has been obtained for the fused iron catalyst, extending for the first time to pressures of 50 psi and 200 psi. Complete reduction of the large amount of data has not yet been completed, however several preliminary conclusions are available. In fitting the kinetic data to a Langmuir-Hinshelwood model, inhibition by water vapor is clearly evident. Over a wide range of conversion the rate is nearly zero order in CO , suggesting it is strongly adsorbed on the catalyst surface. The dependence on

hydrogen is very nearly first order. These dependences are consistent with the kinetic expression reported by Dry as is the inhibition by water. Since the inclusion of water inhibition in a kinetic model requires an additional parameter to be determined, the most useful data would be that from operation under conditions where water vapor partial pressures are low. This suggests the use of low H_2/CO feed ratios to retard the water gas shift reaction.

In our last report we mentioned that an important question in Fischer-Tropsch synthesis is the extent to which α -olefins, the primary products, may be reincorporated into new growing hydrocarbon chains, since this can have a major effect on the overall product distribution. Dwyer and Somorjai (J. Catalysis, 56, 269 (1979)) have presented data that indicated that incorporation of ethylene or propylene into growing chains was a significant secondary reaction on an iron catalyst that markedly affected the value of α in the Flory-Schultz distribution. Our studies under intrinsic kinetic conditions show no effect of degree of conversion on α , which indicates that under our conditions the phenomenon does not occur.

We have now tested this hypothesis further by runs in which ethylene has been added to the synthesis gas. No effect on α was observed over the range of conversions obtained, but some of the ethylene was hydrogenated to ethane. However, it is possible that at low partial pressures of CO , adsorption and reaction of the ethylene may occur. In a CSTR, such as ours, this may be readily obtained by running at high CO conversions. A second run of this type will be conducted in the near future.

B. Mass Transfer Studies

In order to better characterize the mass transfer aspects of the Fischer-Tropsch reaction in a slurry reactor, we plan to examine the effect of solids (density, particle size, loading) upon the mass transfer in gas/liquid stirred systems.

Currently, very little information is available concerning the interaction of the solids with the gas dispersion in a reaction setup such as ours. An approximation that can be made is to combine the solid and liquid phases into one pseudo liquid phase. Attempts can then be made to correlate changes in either of the individual phases with changes in the pseudo liquid phase. Most notably, Joosten (CES, 32, 563 (1977)) has made a preliminary attempt at relating solids loading to apparent viscosity for measurements of the effects of solids loading upon the mass transfer coefficient ($k_L a$) in a stirred contactor.

This treatment is not wholly adequate, since enhancement or reduction of mass transfer due to the presence of solids is not predicted by a model such as this. Additionally, the data available on mass transfer in three phase systems in the literature are primarily restricted to aqueous solutions as the liquid phase. Due to the high surface tension of water relative to organic liquids, the interfacial area measured in aqueous solutions is highly dependent upon such factors as contaminant concentration and ionic strength. The inference of interfacial areas in organic systems from aqueous ones is not clear, which places significant limitations upon scale-up for mass transfer controlled reactions in stirred contactors.

Mass transfer in gas-liquid systems can be measured by either of two general methods: (1) measuring the rate of stripping of a slightly soluble gas (e.g. helium) from the saturated liquid by a

second gas (e.g. nitrogen) (see e.g. Joosten, et al, Chem. Eng. Sci., 32, 563 (1977)) or by (2) a chemical method such as the sulfite method. These methods in general, as applied to aqueous systems, have been recently reviewed by Van't Riet (I.E.C. Process Des. Dev., 18, 357 (1979)). Our preliminary analysis of the physical stripping method indicates that very fast and accurate responses would be needed, in our system. We are currently examining the possibilities of a method published by Sridharan and Sharma (CES, 31, 767 (1976)) who appear to have developed a chemical method for measuring interfacial areas in nonaqueous systems by reaction of carbon dioxide with various amines. This is analogous to the sulfite method used in determining interfacial areas in aqueous systems. Because the method applies to organic liquids, results should be much more relevant to the hydrocarbon synthesis, liquids encountered in Fischer-Tropsch, than would be studies in aqueous media.

We plan to try Sridharan's method in a stirred contactor to measure the effects of solids loading, density and particle size upon gas-liquid interfacial areas. The solids will be nonreactive, but particle loadings, size and density will cover the range used in our experimental Fischer-Tropsch reactor system. The liquid phase initially will be toluene/isopropanol, since data on viscosity, gas solubility and diffusivity are available from the literature on this combination. The gas phase will be carbon dioxide in nitrogen at flow rates comparable to those in our experimental system. Measurements will be made at approximately atmospheric pressure and room temperature.

III. FUTURE WORK

We anticipate that analysis of our intrinsic kinetic data on the fused iron catalyst will be completed during the next quarter, as well as that obtained under mass-transfer controlled conditions. Additional studies will be made with synthesis gas spiked with ethylene under low CO partial pressure conditions to see if incorporation of ethylene into growing chains is significant under these conditions. We also plan to secure analyses (from an outside laboratory) of a representative liquid retained in our autoclave to determine the extent to which high molecular weight material (e.g. liquid C_{20}^+) is formed and whether its formation follows the Flory distribution. We are also planning to make computer simulations of the effects of certain unsteady-state phenomena that may occur in a slurry reactor, such as the effect of the changing liquid composition in the vessel on accumulation or depletion, and on the instantaneous vapor composition.

Future experimental work will be conducted on cobalt and precipitated iron catalysts, conducting extended runs similar to those on the fused iron catalyst.

A Stirred Autoclave Apparatus for Study of the
Fischer-Tropsch Synthesis in a Slurry-Bed

I. Reactor and Trapping Procedures

George A. Huff, Jr. and Charles N. Satterfield

Department of Chemical Engineering

Massachusetts Institute of Technology

Cambridge, MA 02139

Abstract

A mechanically stirred autoclave provides advantages over other types of reactors for fundamental studies of liquid-phase Fischer-Tropsch synthesis, but no information has been published on such use. Particular attention must be paid to obtaining complete suspension of the catalyst, use of non-catalytic materials of construction, and procedures to avoid loss of portions of the wide range of products formed. Many of the aspects of design and operation are applicable to stirred autoclave reactors in general.

Introduction

There has been an upsurge in interest in recent years in the Fischer-Tropsch synthesis and especially in carrying it out in a slurry reactor in which synthesis gas is bubbled into a suspension of finely-divided solid catalyst in an inert liquid, products being volatilized overhead. Industrially the reactor would probably be of a bubble-column or pump-around design and essentially all laboratory and pilot plant studies published to date have been obtained in bubble columns. However, interpretation of results from such studies can be ambiguous in that the complex hydrodynamics makes it difficult to separate intrinsic kinetics from physical effects.

A mechanically-stirred autoclave can be operated so as to behave as a C.S.T.R. with respect to all phases, which simplifies interpretation of results. Although such systems have been used for studying other slurry catalyst reactions, little appears to have been published about significant features in design and operation. When a wide spectrum of products is produced, as here, particular attention must be devoted to trapping and analytical methods. The trapping and analytical procedures developed for our slurry-phase studies discussed here can, of course, also be applied to Fischer-Tropsch vapor-phase studies. Likewise certain aspects of autoclave operation here are applicable to stirred autoclave reactors in general.

Apparatus

A schematic diagram of the flow system is shown in Figure 1. Unless otherwise noted, all components are 316-stainless steel and Teflon. Major components and suppliers are listed in Table I. The unit can operate at pressures to 4 MPa and temperatures of 300°C. Tubing is 0.64-cm o.d. with a wall thickness of 0.089 cm. Unless otherwise noted, manually-operated bellows valves were employed.

Leaks were eliminated by checking all compression tube fittings with a liquid leak detector. Upon pressurizing the system to 8 MPa, a negligible decline in pressure was observed overnight.

Reactor: The synthesis is carried out in a one-liter Magnedrive ("packless") autoclave, illustrated in Figure 2. The reactor has a 7.62-cm inside diameter and an internal height of 24.1 cm. Two baffle bars (0.75-cm wide) are spaced 180° apart. The agitator is a 5.08-cm diameter propeller (3 blades at a 45° pitch), which forces fluid downwards, set above a 5.08-cm diameter turbine impeller. The impeller has 6 flat-bladed disks, each 1.27-cm square, and is 3.5 cm above the vessel bottom.

The autoclave is heated by a two-zone lapped furnace that fits snugly around the unit. The heat input to one zone is preset by a variable transformer, and the amount of heat is regulated on the other zone by a three-mode controller with a digital setpoint which holds the reactor contents within $\pm 0.5^\circ\text{C}$ in the range of 200-300°C, as measured by a type "J" thermocouple located in the thermowell. Since the reactor temperature must be controlled on the skin between the vessel and heating jacket to minimize thermal lag caused by the thick vessel walls, a calibration curve is required between this temperature and that in the thermowell. In our system, for instance, an outside temperature of 315°C is required to maintain the contents at 250°C.

A 10-micron frit was initially installed on the inlet end of the sample tube but it became so easily plugged with either free carbon or finely ground catalyst that it was removed. In order to take a liquid carrier sample, the agitator is turned off and the

contents allowed to settle for a few minutes. In this manner, as little catalyst as possible is removed from the system. The line is then backflushed with helium to purge the tube of its contents back into the reactor. However, liquid carrier samples are taken infrequently to minimize system disturbances.

Gas can be sparged at either of two different locations in the reactor. Assurance that the system is well mixed can be obtained when there is no noticeable change in either activity or selectivity caused by switching from one inlet to the other. Both tubes are open blow tubes with a 0.32-cm i.d., and no operating difficulties have been experienced provided that a slight positive pressure is maintained in the lines. However, check valves are present upstream to guard against incidents.

A 60-micron frit is present at the gas outlet. It gradually becomes plugged over a period of days to weeks, depending on operating conditions, but it is essential for trouble-free operation. Without it a quantity of slurry (e.g. 10-50 cc) may occasionally be suddenly expelled into the outlet lines and wax trap. When this occurs, the reactor and wax trap temperatures suddenly increase 3 to 5°C. This problem appears to be provoked by using H_2/CO ratios in the reactor below one, which probably results in products that have more tendency to foam.

Turbines are reportedly the most effective type of agitator for suspending fast settling, granular solids in a low viscosity fluid (Lyons, 1967), and flat-bladed styles are particularly recommended for gas-liquid dispersions because of their ability to provide a high discharge velocity normal to the gas flow path (Bates et al., 1966). Baffles aid solid suspension in the reactor by directing

flow streams vertically and prevent uncontrolled swirl. Where maximum lifting velocity is required, Lyons recommends that the turbine be positioned directly on the vessel bottom. Unfortunately, the impeller cannot be placed directly on the tank bottom of our autoclave reactor because of its flat-conical shape.

Cold studies in a transparent mock-up revealed that a raised turbine by itself produces the familiar figure-eight flow circulation pattern, but a stirring speed of about 500 RPM was required to suspend a 15-weight percent suspension of 50-micron iron catalyst particles in our system. The addition of the propeller increased vertical circulation and lowered the minimum stirring speed to 200 RPM before we observed any particle settling. In this system, gas flow has a negligible effect on suspension.

Flow Control: The flow of gas is metered by a differential pressure cell which provides a 4-20 milliamper (ma) signal to a controller. At 4 ma there is no flow, and at 20 ma the flow rate is such that there is a pressure drop of 150 inches of water across a jeweled orifice. A calibration curve, accurate to within less than one percent, is easily constructed since flow rate is proportional to the square root of the pressure drop. A new curve is constructed for each different synthesis composition and orifice size. However, in order to avoid recalibrating the flow control system each time reactor pressure is changed, it is isolated from the reactor by a dome-loaded back-pressure regulator which is set at 350 kPa above the maximum working pressure.

A three-mode controller receives the signal from the differential pressure cell and then transmits a 4-20 ma electrical signal to a transducer where it is converted to a 3-15 psig pneumatic output.

This drives an air-operated needle valve in a feed-back control loop. With different valve trims, a wide range of flows, 50 cc/m to 10 L/m for our system, is easily obtained. If desired, the automated flow-metering network can be bypassed by a manually-operated needle valve.

Gas flow rate is measured with soap-film flowmeters which are located downstream of the sample traps. We do not employ a wet-test meter for precise measurements because carbon dioxide and any other soluble gas can dissolve in the water saturator used with it. However, the wet-test meter provides a visual indication of gaseous effluent flow.

Product Sampling: For on-line sampling, it is necessary that the sampling valve be at a temperature sufficiently high to prevent selective condensation or adsorption of products, yet be completely inert. Moreover much water is formed here, which attacks many gas chromatographic packings used for hydrocarbon separation, resulting in a loss in resolution. In earlier work, plugs developed in the stainless-steel tubing which was being heated with electrical heating tapes to $310 \pm 25^\circ\text{C}$ to avoid premature product condensation. Upon examination, the interior wall was found to be heavily coated with a carbonaceous residue. Carbon can be formed rapidly by the decomposition of carbon monoxide to carbon dioxide and carbon with CO/H_2 mixtures over an iron catalyst at temperatures greater than 300°C and elevated pressures (Dry, 1980). The iron in the stainless steel tubing may substantially promote this deleterious reaction. At this time a frit was not being used in the gas outlet so some solids could also have been carried over mechanically from the reactor.

We now condense liquid hydrocarbons and water in two traps, one operated at about 70°C and the second at 2°C (immersed in a water bath that is cooled by mechanical refrigeration). These are termed wax and cold traps (see Figure 3), respectively. This same procedure

as extensively employed in earlier U.S. Bureau of Mines Fischer-Tropsch work with fixed-bed reactors (Anderson, 1956). The melting point of the wax collected in the first trap is about 40-50°C under typical conditions. In our case we use normal octacosane as the liquid carrier in the reactor. This solidifies at 61.4°C and since a slight amount volatilizes and is collected in the wax trap, that temperature is set at 65-70°C to avoid solidification. The cold trap is just above 0°C to prevent water, a major by-product, from freezing in the trap. Both traps are kept as cool as possible to remove the greatest amount of volatile product. In each trap the liquid usually separates into an oil phase which contains the bulk of the hydrocarbon product and an aqueous phase consisting of water plus oxygenated product. These fractions are each analyzed by gas chromatography. Non-condensable gases are analyzed by a dedicated gas chromatograph, using an on-line sampling valve. Procedures are described in Part II of this paper.

Entrainment separators, such as those manufactured by Wright-Austin Co., were not employed because their high internal surface area could cause a significant amount of product to be retained on vessel walls due to the wettability of the condensed material on stainless steel. This problem is enhanced by the fact that only 5 to 50 grams of material is typically trapped over an 3-12 hour period. In our system, we employ empty cylinders and rely on a 100-fold decrease in superficial velocity to knock out entrained liquid drops. Upon completion of a material balance, traps are purged with a slow flow of helium after removing the bulk of the liquid, to insure that all material has been removed.

The system must be at steady-state for this trapping procedure to work effectively as the dead volume in the traps (approximately 300 cc for sample collectors and 1.0 L for bypass) greatly increases the average residence time of gases through the system. Typically the reactor is only half full of slurry, so it also has a gas dead volume. If we simply assume that all three volumes are 300 cc

(autoclave and cold and wax traps) and the system flow rate is 0.2 L/m at reaction conditions, then the holding time for each vessel is 1.5 minutes. Hence, if the concentration of gases above the liquid surface in the autoclave were to experience a step-change, then the rate of change in concentration in the exit stream from the last vessel can be readily calculated, assuming well-mixed conditions in each vessel and neglecting hold-up volume of tubing. See, for example, Denbigh and Turner (1971). For the case above, it would take 12.6 minutes for 99% of a change to be observed at the effluent of the cold trap, or 9.4 minutes for 95%.

The short line of tubing that connects the wax trap and reactor is fabricated of Alonized stainless steel. Alonizing is a process that diffuses aluminum into the stainless steel inner walls so as to render them inert for reactions such as those associated with the Fischer-Tropsch synthesis. Periodic inspection reveals that it remains clean during the course of a run even though it is kept at 300°C. After removal of waxes and heavy hydrocarbons in the first trap, the tubing between the wax and cold traps need only be heated to 100°C to avoid condensation and loss of lighter products on walls. Lines are heated by heavy insulated heating tapes which are suitable for direct contact with metal and temperature-regulated by variable transformers. We have not observed any carbon deposition on stainless steel tubing at 100°C. Furthermore, synthesis gas to which ethylene had been added underwent no secondary hydrogenation either on this tubing, or in the reactor filled with octacosane but without catalyst.

One might expect that C₅ and heavier hydrocarbons would be effectively condensed in the cold trap at 2°C. However, this is not the case, as illustrated in Figure 4. This shows (lower curves),

for a representative run, how the portion of the product of each carbon number is distributed into residual gas, cold trap and wax trap. Note that a significant portion of the C_5-C_{10} fraction remains in the gas phase. The dip at C_2 is because a substantial fraction of the C_2 product is ethanol. The upper curve shows the weight fraction of the total product present in the form of each carbon number. For an accurate material balance it is necessary to combine information on quantities of materials in the three categories and to have accurate analyses.

A material balance on the run shown indicated that 98.7% of the material was accounted for on a weight basis. At 790 kPa and 230-270°C, 1 to 3 weight-percent of the product is high molecular weight material which remains in the liquid carrier, and is not included. For this set of conditions, C_{20} and higher carbon numbers are significantly retained in the liquid due to their lowered volatility. Carbon, formed by decomposition of carbon monoxide via the Boudouard reaction, would also remain in the reactor. The amounts formed in this run appeared to be negligible from a material balance point of view, but this might not be the case for other conditions such as use of very low E_2/CO ratios.

Reduction Unit: The reduction temperature of 400-425°C required for a fused-iron catalyst is too high for the catalyst to be reduced in the reactor itself. Hence, the catalyst is prereduced in a separate vapor-phase unit depicted in Figure 5. The reduction tube is constructed of type-316 stainless steel and measures 1.6-cm i.d. by 40-cm long. Porous frits in the tube ends prevent carry-over and loss of the catalyst. The first 1/4 to 1/3 of the tube is filled with 0.48-cm diameter alumina balls to preheat the gas. An aluminum sleeve fits securely around the stainless steel tube to promote heat transfer and minimize axial

temperature gradients. This fixed-bed reactor is located in a Lindberg tubular furnace (Model 55035).

Reduction of the iron catalyst is carried out with prepurified hydrogen that is first passed through 13-X molecular sieve and activated carbon filters. Flow is maintained by a Fischer-Porter rotameter/needle valve assembly (Model 10A1337). Two 2.5-cm i.d. by 25-cm long Plexiglass tubes, packed with moisture-indicating, 8-mesh Drierite, are located downstream of the reduction unit. As water is a by-product in the reaction of iron oxide with hydrogen, the extent of reduction is followed by observing the rate that the Drierite changes color down the tube (from blue to violet). Upon complete reduction, the catalyst is cooled to room temperature and then transferred, under an inert helium blanket, to the autoclave reactor where it is introduced by a side port in the cover.

Safety: All gas cylinder connections are protected by spring-loaded pressure relief valves. The reactor is located inside a ventilation hood. All exhaust lines of potentially flammable gases are protected by flash arrestors. The synthesis gas feed and reactor furnace are automatically shut down in the event that:

- (1) Carbon monoxide concentration in the laboratory air exceeds 50 ppm (O.S.F.A. limit).
- (2) Reactor temperature, gas feed rate or autoclave pressure substantially deviate from their set-point.
- (3) Electrical power failure.
- (4) Ventilation of reactor off-gases fails.

In an emergency, a solenoid valve is opened (requires power to close) which bleeds off the air on a three-way valve. This rotates, causing the synthesis gas flow to shut off, and turns on a helium bleed through the system.

REACTOR OPERATION

Synthesis Gas: Hydrogen (prepurified) and carbon monoxide (technical grade) are blended to our specification and purchased from Matheson Gas Products, Inc. The synthesis gas is passed through a 13-X molecular sieve and activated carbon traps in the experimental unit followed by a 2-micron filter.

Liquid Carrier: We employ normal-octacosane, $n\text{-C}_{28}\text{H}_{58}$, of 99%+ purity, synthesized by Humphrey Chemical Co., as the slurry liquid. It is a high boiling point (429°C at atmospheric pressure), relatively inexpensive paraffin that is characteristic of waxes produced by the Fischer-Tropsch synthesis. Product analysis is simplified by the use of a pure carrier. The major impurity is bromotetradecane as the paraffin is made by coupling via the Wurtz reaction. We further purify the octacosane by solvent extraction, adding one part paraffin to ten parts tetrahydrofuran (by weight), agitating, and filtering with a Büchner funnel. The material is thoroughly dried before placing it (typically 0.5 L) into the reactor. Octacosane is thermally stable at temperatures below about 325 to 350°C, and it is not hydrocracked in contact with an iron catalyst under reaction conditions so long as carbon monoxide is present with the hydrogen.

Catalyst: We presently employ a fused-iron ammonia synthesis catalyst (from United Catalysts, Inc. and designated C-73). On an unreduced basis, it contains 2.0-3.0% Al_2O_3 , 0.5-0.8% K_2O , 0.7-1.2% CaO , and < 0.4% SiO_2 . It is crushed to particle sizes smaller than 45 microns (325 A.S.T.M. mesh) and pre-reduced with hydrogen at 400-425°C for 72 hours at atmospheric pressure and space velocity of 5000 cc gas/cc catalyst-hr. It is then slurried with the octacosane. We typically employ a 10-15 weight-percent suspension, based on unreduced catalyst weight.

Operating Procedure: Continuous runs lasting more than 1000 hours have been performed. Sampling is taken over a 6 to 12 hour period. After a change in operating conditions, about 6 hours is allowed to elapse before the next sampling period. Since material in the traps forms an aqueous and oil layer, they are separated with a separatory funnel and weighed. The reactor operates continuously 24 hours per day without interruption. Possible change in catalytic activity or selectivity is monitored by periodically repeating a standard run. Analytical procedures are described in Part II. Experimental results showing the change in performance upon the onset of mass-transfer limitations and intrinsic kinetics with an iron catalyst are published elsewhere (Satterfield and Huff).

Blank runs at low space velocities and representative temperature and pressure (250°C and 790 kPa) with the reactor half-filled with liquid but without added catalyst showed that no reaction occurred. This establishes that the walls of the reactor and hot exit tubing were effectively inert and that homogeneous reaction is negligible.

ACKNOWLEDGEMENT

We appreciate preliminary discussions with Dr. R. J. Madon of Exxon Research and Engineering Co., Linden, N.J. This work has been supported by the U.S. Department of Energy and the National Science Foundation.

LITERATURE CITED

- Anderson, R.B., in P.H. Emmett, Ed., "Catalysis IV", Chapter 2
1956, Reinhold, New York.
- Bates, R.L., Fondy, P.L., Fenic, J.G., in V.W. Uhl and J.B. Gray,
Eds., "Mixing - Theory and Practice I", Chapter 3 1966,
Academic Press, New York.
- Denbigh, K.G., Turner, J.C.R., "Chemical Reactor Theory", Second
edition 1971, Cambridge, Eq. 5.16 on p. 79.
- Dry, M.E., Hydrocarbon Processing February 1980, 92.
- Lyons, E.J., in V.W. Uhl and J.B. Gray, Eds., "Mixing - Theory and
Practice II", Chapter 9 1967, Academic Press, New York.
- Satterfield, C.N., Huff, G.A., Jr., a. J. Catal., submitted;
b. Ind. Eng. Chem. Process Design Develop., to be submitted.

Table I. Major Components and Suppliers

<u>Component</u>	<u>Manufacturer</u>	<u>Specification</u>
Differential Pressure Transmitter	Rosemount, Inc.	Model 1151DP for flow with a 150-inch water span and 4-20 ma output in conjunction with Foxboro manifold and jeweled orifice.
Air-Operated Control Valve	Badger Meter, Inc.	Air-to-open, Teflon-packed, research control valve with a 3-15 psig diaphragm, 1/4-in. stainless steel body, and type BLRA positioner.
Flow Controller	Foxboro Co.	Model 62H-5E-OJ with 15-1000 ϕ prop. band, 0.015-3.0 min. reset and 0.015-3.0 min. derivative.
Voltage-to-Pressure Transducer	Fairchild Industrial Products	Model T5120B-44 that proportionally changes a 4-20 ma signal to 3-15 psig air.
Autoclave	Autoclave Engineers, Inc.	Stainless steel, 1-liter autoclave with Magnedrive, cooling coils and 2-lapped zone furnace - Model AFP-1005.
Furnace Temperature Controller	LFE Corp., Process Control Division	Model 238 for a type "J" thermocouple
Mechanical Refrigeration Unit for Cold Trap	Blue M Electric Co.	Model PCC-13A-3
Manually-Operated Needle Control Valve	Chatham Precision, Inc.	Model 10G with an orifice coefficient of 0.0005.

FIGURE CAPTIONS

- Figure 1 Slurry-Bed Reaction Unit: A Pressure Regulator, B Filter-Drier, C Differential-Pressure Cell, D Pneumatic Control Valve, E Manually-Operated Needle Control Valve, F Dome-loaded, Back Pressure Regulator, G Condensed Products, H Electric Furnace, I Thermocouple Well, J Baffle, K Magnedrive Stirrer, L Pressure Gauge, M 300 cc Trap, N 1.0 L Trap, O Gas Sample Valve, P Soap-Film Flowmeter, Q Water Saturator, R Wet-Test Meter, S Slurry Sample Tube, T Agitator, U Three-way, Air-Operated Valve, V Mechanical Refrigeration Unit, W Strip Heater
- Figure 2 One-Liter Autoclave Reactor
- Figure 3 Schematic of Liquid Trap
- Figure 4 Iron Catalyst at 269°C and 790 kPa. H_2/CO ratio at inlet = 1.3. H_2/CO ratio at outlet = 9.4.
- Upper curve: Distribution of Product by Carbon Number, N
- Lower curves: Distribution of Product of Carbon Number, N, into Samples by Volatility.
- Figure 5 Reduction Unit: A Pressure Regulator, B Filter-Drier, C Manually-Operated Needle Control Valve, D Rotameter, E 2-Micron Porous Frit, F Aluminum Jacket, G Electric Tube Furnace, H Thermocouple, I Columns Packed with Drierite, J Water Saturator, K Wet-Test Meter, L 3/16-in. Alumina Balls

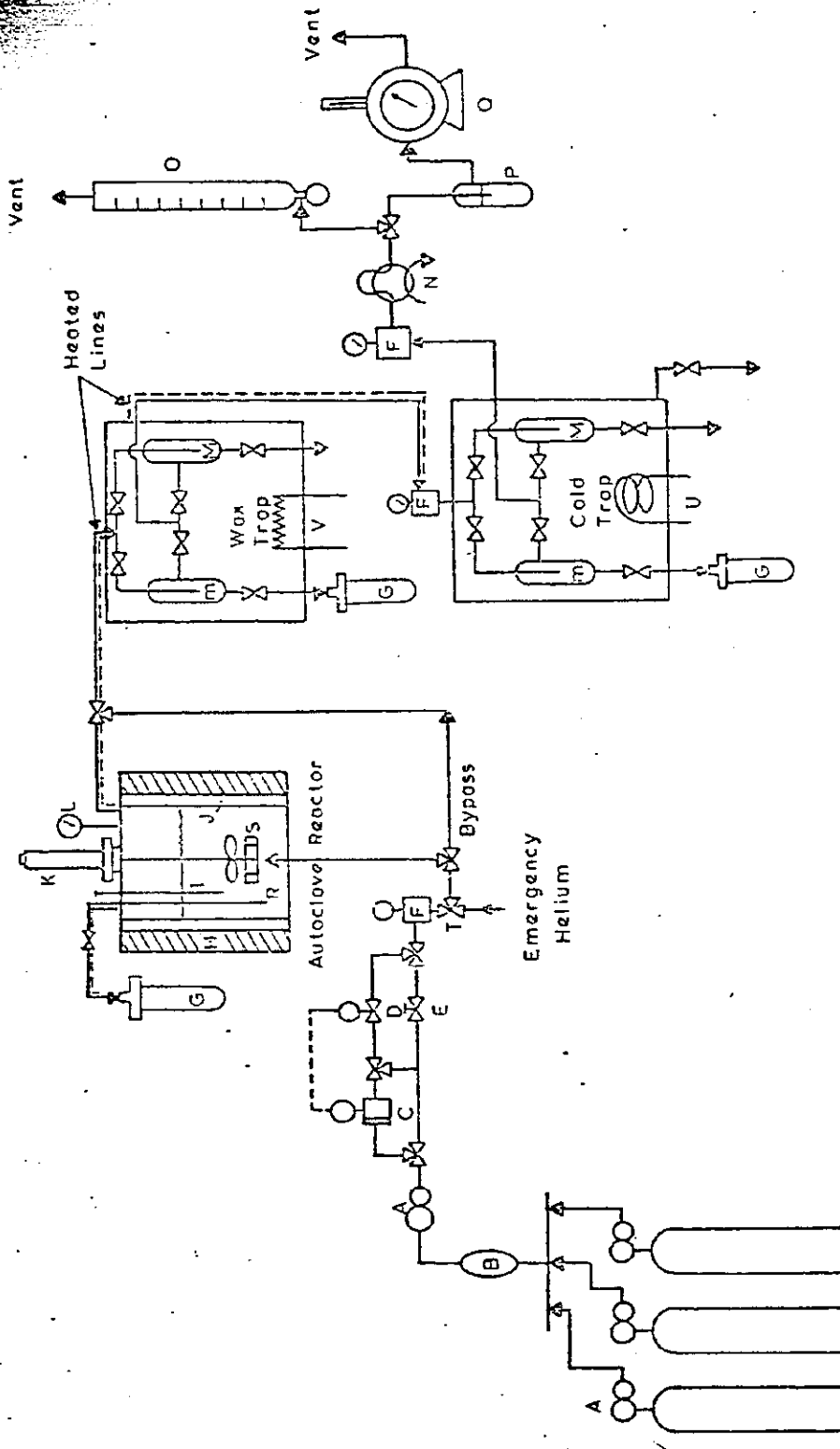


FIG. 1

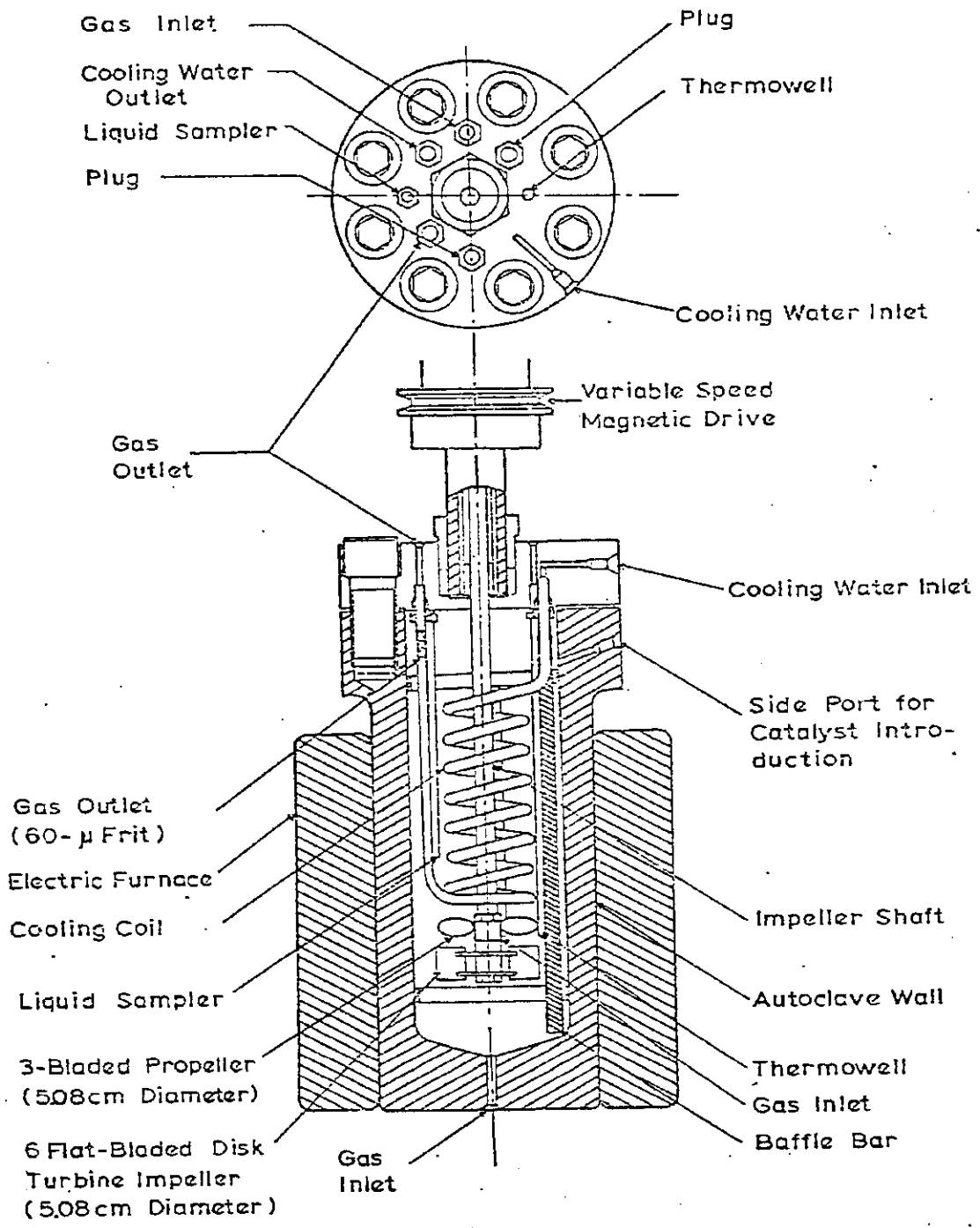


FIG.2

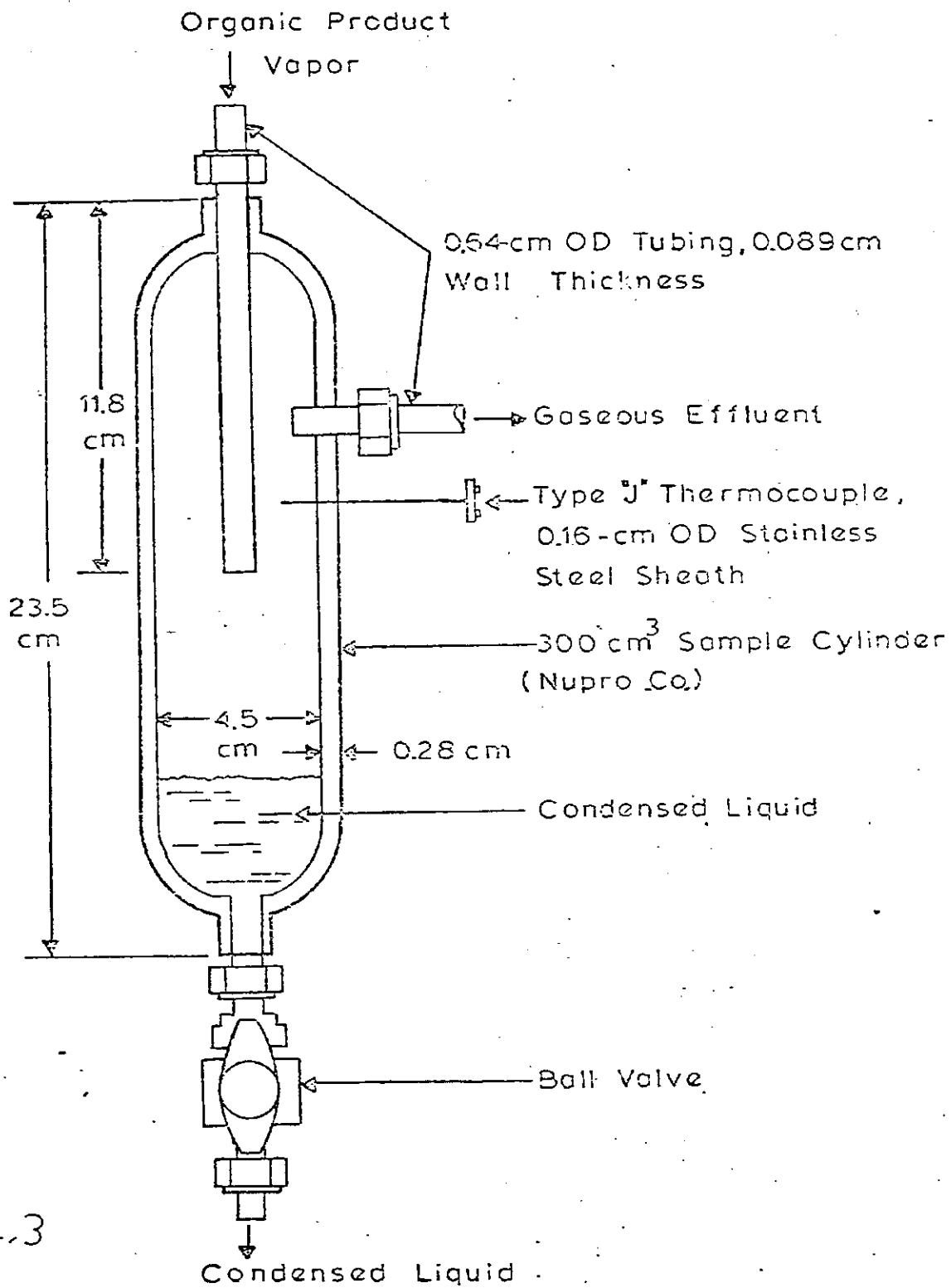


FIG. 3

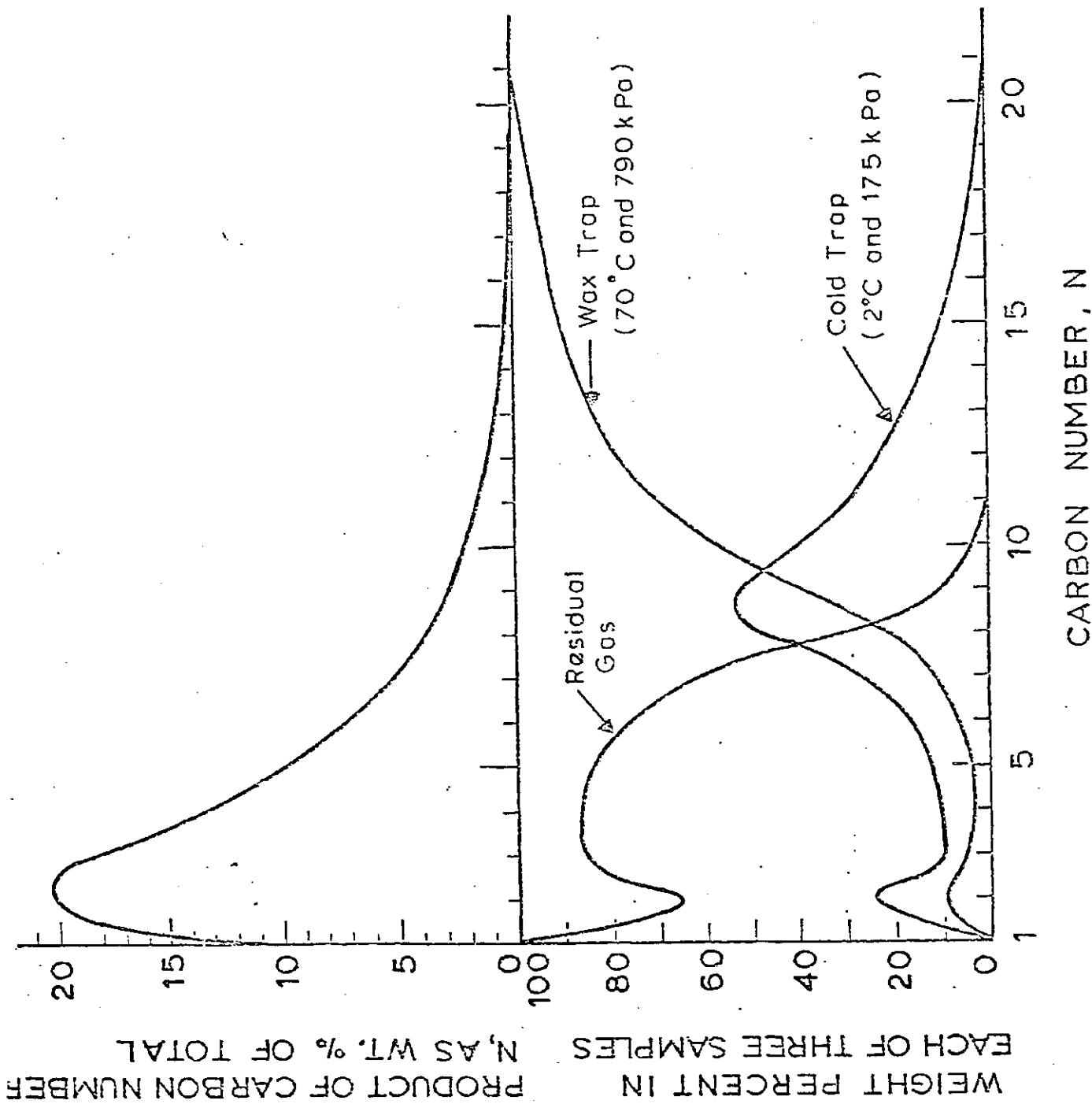


FIG. 4

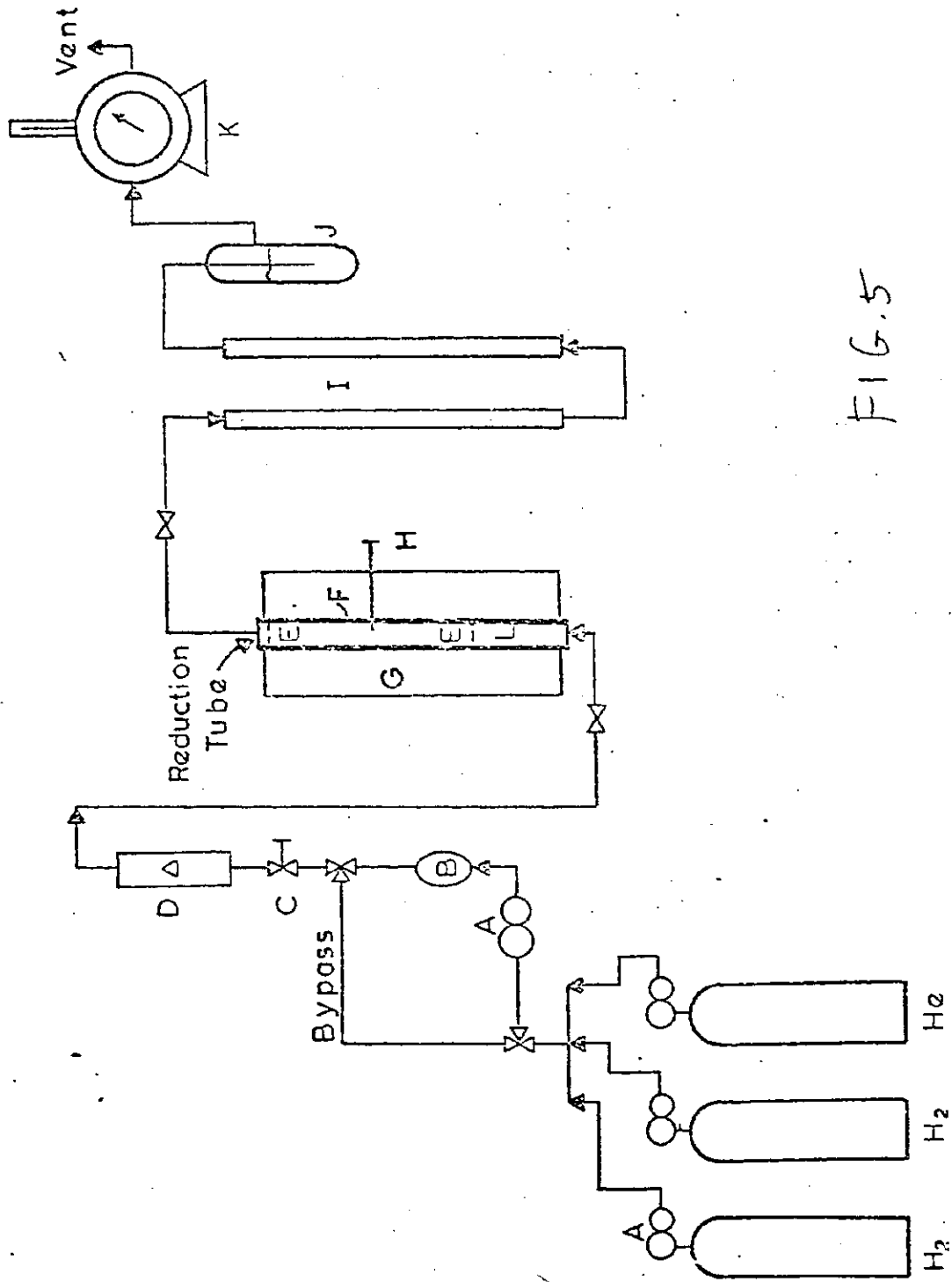


FIG. 5