ANL-76-47

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PHYSICAL PARAMETERS IN SYNTHOIL PROCESS

Quarterly Report for the Period
January—March 1976

by

J. Fischer, R. Lo, S. Nandi,
D. Fredrickson, K. Javdani, T. Cannon,
T. Bump, T. Mulcahey,
H. Huang, and A. Jonke

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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

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9700 South Cass Avenue
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Chemical Engineering Division

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A development program is being carried out to obtain information applicable to the SYNTHOIL process for converting coal to liquid fuel of of low sulfur content. This report presents information on (1) the design of apparatus for measuring coefficients of heat transfer from SYNTHOIL process feed and effluent products to process vessel walls, (2) tests on the removal of solids from oil produced in coal liquefaction processes, (3) the design of a test unit for evaluating new catalysts for the SYNTHOIL process, and (4) proposed work on the flow properties and mass transfer characteristics of gas-liquid-solid mixtures during upward transport through a cylinder.

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Coal
Calcium Sulfates
Additives
Ashes
Desulfurization
Liquefaction
Fuel Oil
Residual Oils
Sulfur
Catalysts
Hydrogen
Calorimeter
Heat Transfer
Flocculants
Viscosity
Surfactants
Particle Size Distribution
Three Dimensional Flow
Mass Transfer
Hydrogenation
Slurries

17b. Identifiers/Open-Ended Terms

Air Pollution Control
SYNTHOIL
Petrowet R
Alkanol WXN

17c. COSATI Field/Group

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PHYSICAL PARAMETERS IN SYNTHOIL PROCESS

by

J. Fischer, R. Lo, S. Nandi, D. Fredrickson, K. Javdani, T. Cannon, T. Bump, T. Mulcahey, H. Huang, and A. Jonke

ABSTRACT

This work is in support of the development of processes for converting coal to liquid fuel of low sulfur content and suitable for use in power production. Most of this effort is intended to produce information applicable to the SYNTHOIL process.

In the SYNTHOIL process for converting coal to a low-sulfur fuel oil, coal is liquified and hydrodesulfurized in a turbulent-flow, catalytic packed-bed reactor. A slurry of coal in recycled oil is reacted with hydrogen at 450°C and 2,000-4,000 psi in the presence of Co-Mo/SiO_2-Al_2O_3 catalyst. Turbulent flow of fluid prevents the coal's mineral matter from settling and plugging the reactor. The gross liquid product made in a 1/2 ton per day pilot plant at PERC is centrifuged to remove the unreacted solids producing a low-sulfur, low-ash liquid fuel.

The work for this program includes tasks I-IV started in September 1975 and a new task V started in January 1976.

<u>Task I, Heat of Reaction of Hydrogen with Coal Slurries</u>
Determination of the heat of reaction of coal-oil slurries with hydrogen, in the presence and absence of catalysts, at 2,000-4,000 psi and 400-475°C. A contract has been awarded to a manufacturer for the design and construction of the calorimetric system.

<u>Task II, Heat Transfer Coefficient</u>
Determination of the coefficients of heat transfer from SYNTHOIL reactor fluids to heat exchangers in the following ranges of conditions: pressure, 2,000-4,000 psi; temperature, to 465°C. Design of the test unit is 90% completed.

<u>Task III, Additives to Facilitate Separation of Solids from Liquids</u>
Investigation of additives to facilitate the removal of solids from oil produced in coal-liquefaction processes. Additives being considered are those which would act as flocculating agents and/or would alter the physical properties of the mixture, improving separation in subsequent steps. The viscosities of SYNTHOIL products were reduced when two DuPont surfactants were individually added. A flocculation-test system has been assembled, and a small precoat filtration unit is being constructed. Also, the particle-size distributions of the benzene-insoluble fraction and ash contents of SYNTHOIL products were determined.

Task IV, Catalyst Testing

Testing of new catalysts in 300-hr runs in a continuous high-pressure coal-liquefaction unit to operate at 2000-4000 psi and 450°C at a minimum feed rate of 4 oz/hr of coal slurry containing 35-50% coal. The product samples will be collected and analyzed for: coal conversion; sulfur, nitrogen, and oxygen removal; hydrogen consumption; and the viscosity and specific gravity of the product oil.

The design of the test unit and ordering of all long-delivery-time components have been completed. A contract for the construction and installation of the test unit has been awarded. The unit will be delivered to us in June 1976. Installation and final testing of the test unit will be completed by the end of August 1976.

Task V, Transport Properties in Multiphase Flow

Determination of the flow and mass transfer characteristics in the upward flow of gas-liquid-solid mixtures in cylindrical tubes. Flow characteristics studies in phase 1 of this task will include the identification of flow patterns and the measurement of pressure drop, phase holdup, and phase distribution as a function of flow rate and physical properties of the phases.

Phase 2 will include the measurement of solid-liquid and gas-liquid interphase mass transfer rates for the different flow configurations.

SUMMARY

Heat of Reaction of Hydrogen with Coal Slurries
(S. Nandi and D. Fredrickson)

The objective of this task is to obtain heat-of-reaction data for the hydrogenation of coal slurries typical of those used in the SYNTHOIL process under SYNTHOIL process conditions. The heat release data is required for proper design of the slurry preheater and the reactor of the SYNTHOIL plant.

To accomplish this task, the reaction of hydrogen with a slurry consisting of coal and process-derived oil will be carried out in a calorimetric pressure vessel at 2000-4000 psi and temperatures up to 475°C. A contract has been awarded to Calorimetrics of Boulder, Colorado, for the design and building of this calorimetric system. A functional design meeting has been held with Calorimetrics and design drawings were reviewed.

Heat Transfer Coefficient
(T. P. Mulcahey and T. R. Bump)

The objective of this task is to provide measured coefficients of heat transfer from SYNTHOIL process feed and effluent products to container walls, for use in the future design of heat transfer equipment for the process. Preliminary unit configuration and component sizing of a test unit were completed, based on the slurry and hydrogen flow rates being comparable to those in a 1/2 ton/day plant, *i.e.*, 25 lb/hr of slurry feed and 1300-scfh hydrogen flow. Unit design, except for the test module, has been completed and long-lead items have been ordered. Specifications were prepared for procurement of the unit and a vendor has been selected. Design of the test module is progressing; the detailed design of the straight and coiled elements is complete and design of the heating elements and mixers is under way. An error analysis was initiated that will help in the selection of test module instrumentation and control equipment.

Additives to Facilitate Separation of Solids from Liquids
(H. Huang and S. Nandi)

The objective of this task is to investigate the effect of additives in facilitating the removal of solids from oil produced in coal liquefaction processes. Additives being considered are those which would act as flocculating agents and/or would alter the physical properties of the mixture, improving separation in subsequent steps.

A dozen commercially available chemicals have been individually added to the gross product (uncentrifuged) from the SYNTHOIL pilot plant to determine their individual effects on reducing the viscosity of the gross product. We observed that two DuPont surfactants,

Petrowet R and Alkanol WXN (trade names) are capable of reducing the viscosity of the uncentrifuged oil, at 83°C--by 11% and 14%, respectively--when added to the oil at about 1 wt %. We also observed that the viscosities of the SYNTHOIL (centrifuged) product and the benzene-soluble fraction of the gross product can be reduced by 12% and 15%, respectively, with the addition of about 1 wt % of the Petrowet R. Identification of the mechanisms responsible for the reduction in viscosity should be helpful in identifying effective additives and is currently being worked on.

We have found that simple conventional flocculation evaluation tests that are used in sewage treatment are not suitable for the SYNTHOIL gross product, and have therefore adopted a sample-analysis technique to assess the ability of additives to induce flocculation of SYNTHOIL gross product. Assembly of the test unit is complete and evaluation of the additives for SYNTHOIL gross product will start.

A small precoat filtration unit is being built to further evaluate promising additives found in viscosity reduction and/or flocculation tests. These studies can provide information needed in designing a large-scale filtration system.

In other work, the particle-size distributions of (1) the benzene-insoluble fraction of the gross product, (2) the ash from the gross product, and (3) the ash from SYNTHOIL product were measured, using a Coulter counter. The median particle sizes were 12 μm, 7 μm, and 6 μm, respectively.

Catalyst Testing
(R. Lo, T. Cannon)

The objective of this task is to evaluate new commercially available catalysts for use in the SYNTHOIL process. A continuous high-pressure coal liquefaction unit will be constructed to operate at SYNTHOIL process conditions--450°C and 2000 to 4000 psi. Catalysts will be tested in 300-hr continuous runs. The evaluation will be made on the basis of analyses of coal input and product samples. Samples collected at least once every 24 hr will be analyzed to obtain information on: percent coal conversion; sulfur, nitrogen, and oxygen removal; hydrogen consumption; and the viscosity and specific gravity of the gross liquid product.

Bids for the construction and installation of the Catalyst Test Unit were received and a contract has been awarded, effective February 2, 1976. We expect the unit to be delivered to us by June 1976, and expect installation and final testing to be completed by the end of August 1976. The recycle gas compressor, the high-pressure wash-water pump, and the preheater and reactor furnaces, which are components of the Catalyst Test Unit, have been received. Delivery of all other long-delivery-time components except the pressure vessels by the end of April is expected. Delivery of the high pressure vessels is scheduled for mid-June.

Work on preparation of the laboratory facility for the Catalyst Test Unit is about 70 percent complete.

Transport Properties in Multiphase Flow*
(K. Javdani,** T. Cannon)

The general objective of this research program is to investigate the flow properties and mass transfer characteristics of gas-liquid-solid mixtures during upward transport through a cylinder. The information sought is needed for the analysis of the heat transfer coefficient data to be obtained in Task II and for the correct application of the data to the design of SYNTHOIL pilot plant heat exchangers. This program will consist of two phases. Flow characteristics of gas-solid-liquid mixtures will be studied in phase 1, and the interphase mass transfer will be studied in phase 2.

Study of the flow characteristics in phase 1 will include determination of the conditions that produce different flow patterns. Boundaries that separate regimes of different flow patterns will be determined by measuring pressure drop. Phase distribution and phase holdup will also be determined. The results will be correlated in terms of the velocity of each phase and its physical properties such as density, viscosity, and surface tension. The effects on flow properties of particle size and particle concentration in the slurry will also be determined.

The study of interphase mass transfer that will be pursued in phase 2 will include measurement of the mass transfer rate from gas to liquid, as well as from solid to liquid, under the flow conditions most likely to be found in a preheater of the SYNTHOIL process. The effects on the mass transfer coefficient of particle size and concentration in the slurry and the physical properties of the fluids involved will be determined. This phase of the investigation will start after preliminary results have been obtained on the flow characteristics. For this reason, the work related to phase 1 is emphasized in initial work.

An experimental unit has been constructed for phase 1 of this investigation. Experiments are being conducted at ambient conditions. To simulate the properties of the fluids in the actual SYNTHOIL process, mixtures of either of two white mineral oils that have different viscosities and helium or nitrogen are used as the working fluids. Preliminary experimental results indicate that the experimental unit is capable of providing the required data.

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*Proposed new task.

**Research Associate, Argonne National Laboratory, and Assistant Professor of Chemical Engineering, Aryamehr University of Technology, Tehran, Iran.

Schedule for Tasks I to V

A breakdown of each of the five tasks into various phases of work is given in Table 1.

Table 1. Schedule for Tasks I to V

Task	Description	Start	Complete
I	Design, construct, and shake down heat of reaction calorimeter	9/75	10/76
	Determine the heat of reaction slurry with hydrogen (with catalyst absent)	11/76	3/77
	Determine heat of reaction of slurry and hydrogen in presence of catalyst	4/77	9/77
II	Design, construct, and shake down heat transfer coefficient test loop	9/75	11/76
	Design and construct test section	10/75	9/76
	Determine film coefficient for cooled effluent	12/76	3/77
	Determine film coefficient for heated feed	4/77	9/77
III	Determine viscosity and temperature coefficient of coal-liquefaction products with additives	12/75	10/76
	Determine flocculation characteristics of coal liquefaction products with additives	5/76	6/77
IV	Design, construct, and shake down catalyst test unit	9/75	8/76
	Test four catalysts in 300-hr runs	9/76	8/77
	Test additional catalysts	9/77	7/78
V	Design and build experimental unit	1/76	4/76
	Determine flow characteristics in the flow of gas-liquid-solid mixtures	5/76	6/77
	Determine mass transfer characteristics by measuring interphase transfer rates in solid-liquid and gas-liquid systems	9/76	12/77

I. Heat of Reaction of Hydrogen with Coal Slurries
(D. Fredrickson and S. Nandi)

The objective of this program is to obtain heat of reaction data for the SYNTHOIL process, in which a coal slurry in process-derived oil is converted to low-sulfur fuel oil by catalytic hydrogenation.

In our last report (ANL-76-2), a general description was given of the proposed calorimetric system required to accomplish the objectives of this program. A contract has now been awarded to Calorimetrics of Boulder, Colorado, for the design and building of this calorimeter.

The first part of the contract provides for a functional design review meeting with Dr. E. D. West from Calorimetrics. This meeting was held on March 18-19 and design drawings were reviewed. Steps in calorimeter design and fabrication (Table 2) will be reported subsequently.

II. Heat Transfer Coefficient
(T. Mulcahey and T. Bump)

The objectives of this task are to determine heat transfer coefficients applicable to the SYNTHOIL process feed heat exchangers and effluent heat exchangers and to identify mechanisms for the heat transfer process. Information will be supplied, in a form suitable for SYNTHOIL heat exchanger design, on heat transfer film coefficients for (a) SYNTHOIL reactor effluent cooled by a metal surface and (b) SYNTHOIL feed heated by a metal surface. The following ranges of conditions will be covered:

Pressure:	2,000-4,000 psig
Temperature:	200-475°C
Flow of Fluids:	turbulent

The experimental work will be carried out in a 1/2-ton-per-day heat transfer test loop which will include a slurry makeup system, loop slurry feed tank, feed pump, heater, cooler, letdown tanks, and power, flowrate, and temperature instrumentation. Three different coals suitable for SYNTHOIL processing will be studied in the investigation. Theoretical modeling of the heat transfer process will be attempted, correlating primary interaction of the liquid, particles, and walls. The resulting model should be capable of describing the heat transfer rates, as well as the temperature history of the particles in the slurry.

Preliminary unit configuration and component sizing of a test unit has been completed based on the slurry and hydrogen flowrates being comparable to those in a 1/2 ton/day plant, *i.e.*, 25 lb/hr of slurry feed and 1300 scfh hydrogen flow.

Table 2. Schedule for Heat of Reaction Calorimeter

Activity	1976 Jan	Feb	Mar	Apr	May	June	July	Aug	Sept	Oct	Nov	Dec
Contract Awarded		▲										
A. Design Plan		■	■									
1. ANL Review			▲									
B. Functional Design		■	■									
1. ANL Review			▲									
C. Design, Drawings		□	□	□								
1. ANL Review				△								
D. Technical Program Review Meeting				△								
E. Quality Control Plan		□	□	□								
1. ANL Review				△								
F. Mfg. Plan		□	□	□								
1. ANL Review				△								
G. Design, Fabrication, Pressure Vessel				□	□	□	□	□				
H. Fabrication Cal. Parts				□	□	□						
1. Fabrication Review (Boulder)						△						
I. Assemble Calorimeter					□	□	□	□				
1. Fabrication Review (Boulder)								△				
J. Logic, Timing Circuitry			□	□	□	□						
K. Control, Measuring Compon.				□	□	□						
L. Adapt, Test, Control Compon.								□				
M. Test Entire System									□			
1. Fabrication Review (Boulder)									△			
N. Packaging Plan									△			
O. Disassemble, Pack, Ship									□			
P. Shipping Notice									△			
Q. Reassemble at ANL										□		
R. Inspection, Tests, Records										△		
S. Manuals										△		
T. Shakedown Tests										□	□	
U. Runs, Coal, H_2											□	

Heat Transfer Test Unit

The unit configuration has been expanded to include a slurry makeup system that will allow coal tar-derived products to be blended with coal to form the wetted 93°C (200°F) slurry feed material for the heat transfer coefficient measurement unit. The makeup system capacity will be approximately 140 gal. To ensure timely completion of this system, an additional component has been added to the list of long-lead items; this component is a low-pressure slurry circulation and transfer pump having a circulating capacity of 120 gal/hr. Procurement of the pump was initiated, and it is currently on order.

The specifications for the procurement and assembly of all components of the heat transfer coefficient test unit except the test module have undergone preliminary design and safety reviews and procurement has been initiated. The bids for the test unit have been evaluated and a vendor has been selected. Table 3 shows the proposed schedule for construction of the unit. This schedule will be updated when the vendor's manufacturing plan has been received and approved.

Procurement of Long-Lead Items

In order to expedite the purchase of components and to prevent delays in construction, long-lead procurement items were ordered while work was progressing on unit design. Most of the major long-lead items currently on order were described in the previous quarterly report (ANL-76-2). Scheduled delivery dates for the long-lead items and the description of a new item are as follows:

1. Low-pressure slurry circulation and feed pump for the slurry feed module (50 gph). April 1976

2. High-pressure slurry feed pump. June 1976

3. High-pressure wash-water pump. June 1976

4. High-pressure hydrogen-transfer compressor. April 1976

5. High-pressure hydrogen makeup compressor. April 1976

6. High-pressure slurry receiver. June 1976

7. High-pressure water-wash receiver. June 1976

8. Low-pressure slurry makeup system circulation and transfer pump. This pump is similar to the item 1 pump except that its flow-rate is 120 gph and the delivery pressure is 40 to 60 psi. Delivery of this equipment is expected in April 1976.

Test Module Description

The conceptual design of the test module has been fixed, and no change is expected unless major economic or safety problems are

Table 3. Schedule for Construction, Shakedown, and Testing of the Heat Transfer Coefficient Measurement Unit

Activity	1976								
	Mar	Apr	May	June	July	Aug	Sept	Oct	Nov
Contract Awarded	▼								
System Design and Engineering	—	—	—	—	—	—			
Procurement of Materials		—	—	—	—	—			
Technical Program Review Meeting				▽					
Assembly of System Modules					—	—			
Intermediate Testing of Modules						—	—		
Delivery of System Modules						—	—		
System Integration and Installation							—	—	
System Debugging and Shakedown								—	
System Performance Tests								—	—

encountered. The preliminary detailed design of the cooling elements has been completed and is under review. Cost estimates for the fabrication of the single straight element and three identical coiled cooling elements were requested and were received from the ANL shops. An information package is being prepared for a safety design review of the test module cooling elements. The cooling elements will use room air as the coolant. Each cooling element will have an individual countercurrent and controlled air flow so that the heat loss in each element can be measured and controlled.

There is satisfactory progress on the preliminary detailed design of the individual heating elements and on the slurry temperature monitoring elements to be located at the entrance and exit of each heating and cooling element. A preliminary analysis is being performed to estimate the errors involved with the use of a.c. and d.c. electric heating power. This error analysis includes an assessment of techniques available for minimizing the errors by proper selection and attachment of available instrumentation.

Test Module Instrumentation

It is planned to measure the wall temperature of each 6-ft heating element with thermocouples at 1-ft intervals and to place additional thermocouples on the electrical lead attachments to the elements. Thermocouples also will be mounted on the straight-tube cooling element--on the shell and tube at 1/2-ft intervals. Thermocouples will be mounted on the coiled cooling elements at approximately 3-ft intervals on the 22 ft of tubing; the locations of the thermocouples on the shell will correspond to those of the tubing thermocouples.

The voltage at one lead of each of the thermocouples on the heating element and of the electrical lead attachments to the heating elements will be measured; these measurements will provide information for calculation of (1) the heat input power in each segment of a heating element and (2) possible error corrections for the individual thermocouple outputs to obtain accurate temperatures. Electrical measurements will be made to accurately determine the electrical current flowing in each heating element. The total number of points at which thermocouple and electrical measurements accurately determine the heat transfer coefficient is expected to exceed 180.

III. Additives for Separation of Solids from Liquids (H. Huang and S. Nandi)

The objective of this task is to investigate the use of additives to facilitate the removal of solids from oil produced in coal-liquefaction processes. These additives should be capable of acting as flocculating agents and/or altering the physical properties of the mixture so as to improve separation in subsequent steps.

The present phase of this study is limited to the evaluation of candidate additives, based on their abilities in reducing the viscosity of the mixture and/or inducing flocculation. A small precoat filtration system is being constructed to investigate the effects of promising additives on filtration performance. Also, scouting tests will be conducted to assess the potentials of other schemes for removing the solids from the SYNTHOIL products.

Viscosity Reduction

As mentioned in our preceding quarterly report (ANL-76-2), the viscosity of SYNTHOIL* product changed when it was exposed to air, possibly due to the oxidation of the oil sample and the loss of vapors. To minimize the oxidation effect, we have confined the viscosity-measuring unit in a plexiglass enclosure where it is continuously purged with nitrogen.

A dozen commercially available chemicals have been individually added to the gross liquid (uncentrifuged) product to determine their individual effects on reducing the viscosity of the product oil. Most of the additives, amides or sorbitan esters, had little or no effect on the viscosity of the oil, but some additives significantly reduced the viscosity when only a small amount was added to the oil. Of the additives that induce significant reduction in viscosity, two DuPont surfactants had rather large effects and have been investigated in detail.

Petrowet R and Alkanol WXN are the trade names of these two DuPont surfactants. The former is a solution of sodium dodecyl sulfonate in water and isopropyl alcohol, and the latter is a solution of sodium dodecyl benzene sulfonate in the same solvent. Their properties are listed in Table 4.

Viscosity measurements for the SYNTHOIL gross product, with the addition of various amounts of Petrowet R or Alkanol WXN, were conducted as the samples were heated from 82°C to 120 or 140°C and again at the same temperatures as the samples were cooled. The complete results are tabulated as Table 5. Part of the results obtained when the temperature was increased from low to high are also plotted in Figures 1 and 2. The estimated precision for the percent viscosity reduction values is about 2%.

For both additives, the percentage reduction in viscosity with increasing temperature decreases at higher temperatures and becomes negligible at about 140°C. As can be seen from the results for 0.91 wt % Petrowet R and 1.29 wt % Alkanol WXN in Table 5, when the additive was heated to 140°C, the percentage reduction in viscosity due to the additive was not reversible upon lowering of the temperature--the viscosities obtained were equivalent to those obtained in the absence of additive. Reversibility was only partial when the temperature maximum had been 120°C. These phenomena may be attributed to deactivation of the additives at higher temperature, which may be due

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*Centrifuged product oil from SYNTHOIL pilot plant, Pittsburgh Energy Research Center.

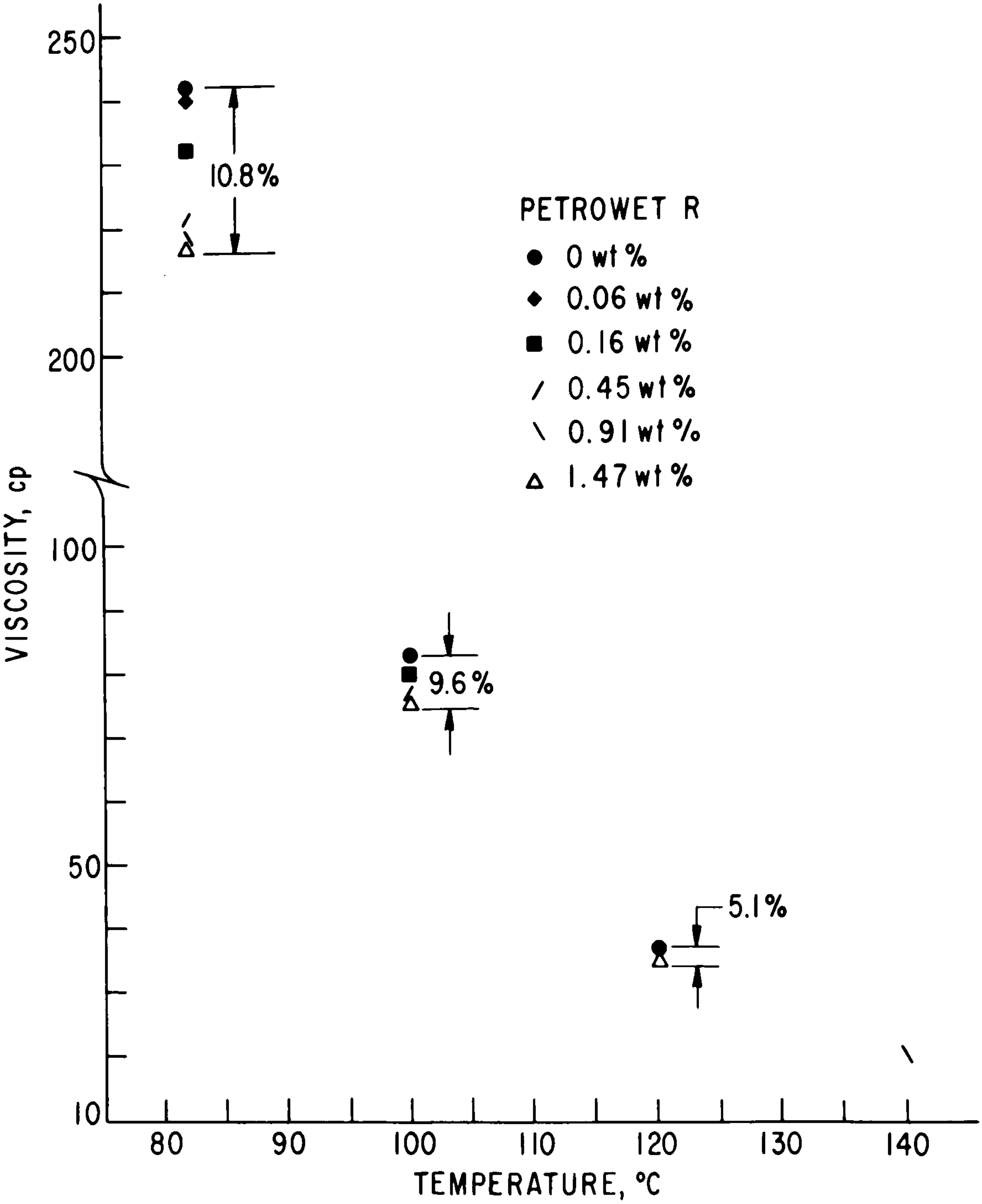


Fig. 1. Additive Effect of Petrowet R on Viscosity of Gross SYNTHOIL Product.

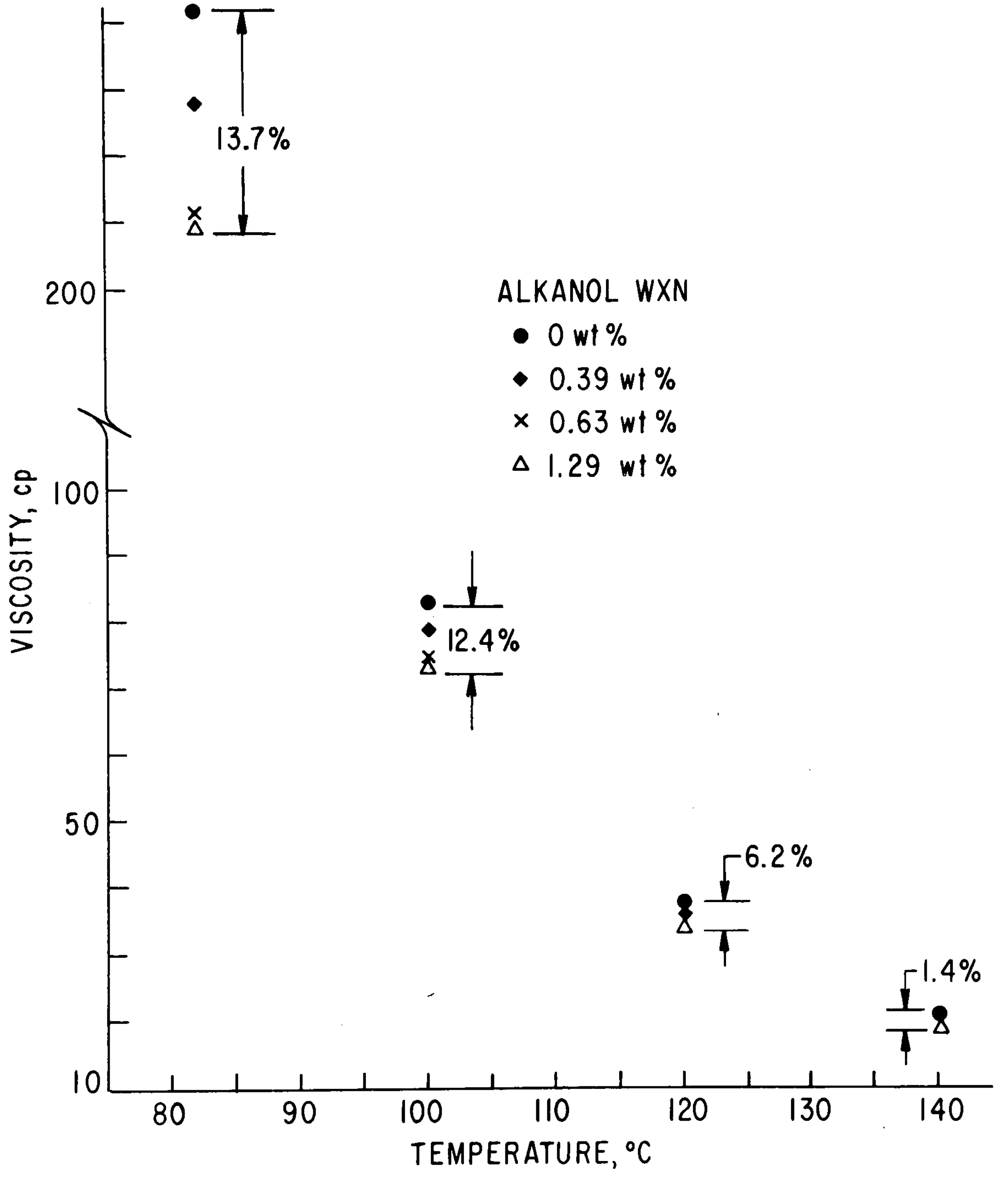


Fig. 2. Additive Effect of Alkanol WXN on Viscosity of Gross SYNTHOIL Product.

Table 4. Properties of DuPont Surfactants

	Petrowet R	Alkanol WXN
Chemical type	Anionic	Anionic
Active ingredient	Sodium-dodecyl sulfonate	Sodium-dodecyl benzene sulfonate
Concentration of active ingredient	22%	30%
pH, 1% of sorbent in water	4.0 to 5.5	7.5 to 9.0
Flash point	91°C	100°C
Density, lb/gal	9.0	8.6
Solubility in H_2O	Miscible	Miscible
Stability		
Acid	Stable	Stable
Alkali	Stable	Stable

to vaporization of solvents in the additives. In order to determine the mechanism for viscosity reduction, we will obtain pure sodium dodecyl sulfonate and investigate its effect on viscosity reduction in the absence of solvent.

The relationship between the percentage reduction in viscosity and the amount of the additive at 82°C is shown in Fig. 3. Apparently, the percentage reduction in viscosity increases linearly with concentration of additive to a certain point, and then the reduction in viscosity is less for further increases of concentration of additive.

It would be helpful in our search for suitable additives if the mechanisms responsible for the reduction of over 10% of the original viscosity (observed when 1 wt % of certain chemicals were added to the product oil) could be identified. Since the gross product from SYNTHOIL (uncentrifuged) contains about 5 wt % ashes and about 12 wt % benzene-insolubles, it has been suggested that the viscosity reduction (observed with a viscometer) is simply a result of the effect of the additives on the suspended solids. That is, the additives may serve as dispersing agents, lowering the interaction forces between suspended solids and liquid and consequently reducing the overall resistance for momentum transfer.

Table 5. Effect of Additives on Viscosity of Gross Product from SYNTHOIL Pilot Plant.

For each concentration, viscosity measurements were taken at increasing temperatures to the temperature maximum and then at descending temperatures.

Additive (wt %)	Viscosity[a] (cp)						
	82°C	100°C	120°C	140°C	120°C	100°C	82°C
0	241.	82.8	37.0	21.3	37.2	85.2	275.
0	242.	83.0	37.4		37.4	83.8	247.
Petrowet R							
0.06	240. (0.8)	82.7 (0.4)	36.8 (1.6)		36.8 (1.6)	83.0 (1.0)	244. (1.2)
0.16	232. (4.1)	80.2 (3.4)	36.2 (3.2)		36.2 (3.2)	81.5 (2.7)	234. (5.3)
0.45	221. (8.7)	76.5 (7.8)	35.7 (4.5)		35.7 (4.5)	80.5 (3.9)	240. (2.8)
0.91	218. (9.5)	75.0 (9.4)	35.4 (4.3)	21.2 (0.5)	37.2 (0.0)	85.4 (0.0)	276. (0.0)
1.47	216. (10.8)	75.0 (9.6)	35.5 (5.1)		35.5 (5.1)	80.5 (3.9)	233. (5.7)
Alkanol WXN							
0.39	228. (5.8)	79.0 (4.8)	36.2 (3.2)		36.2 (3.2)	80.4 (4.1)	240. (2.8)
0.63	211. (12.8)	74.5 (10.2)	35.0 (6.4)		35.0 (6.4)	76.4 (8.8)	228. (7.7)
1.13	209. (13.6)	73.2 (11.8)	34.8 (7.0)		34.8 (7.0)	76.6 (8.6)	228. (7.7)
1.29	208. (13.7)	72.5 (12.4)	34.7 (6.2)	21.0 (1.4)	37.0 (0.5)	84.5 (0.8)	274 (0.4)

[a] Percentage reductions in viscosity from original viscosities are in parentheses.

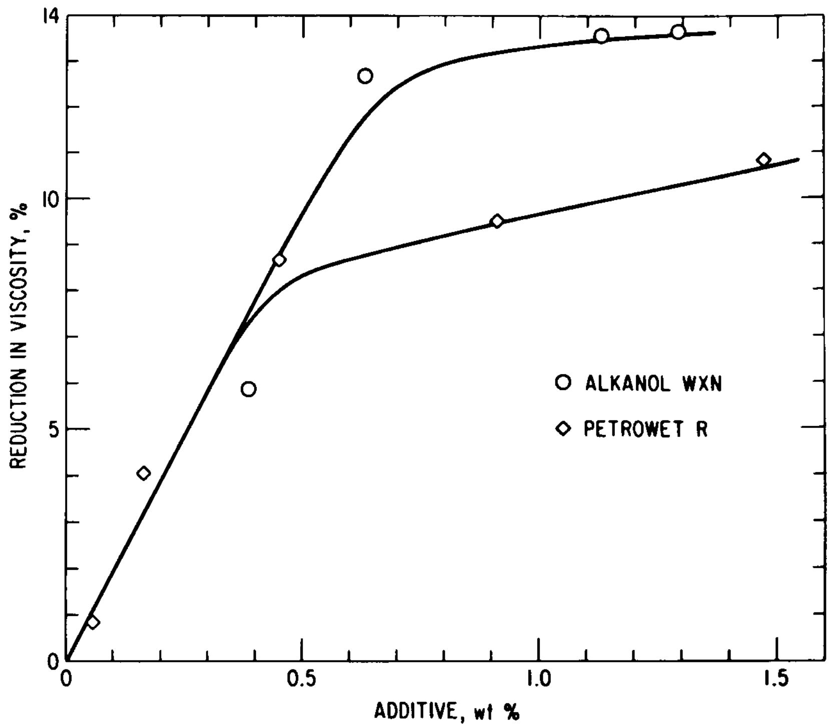


Fig. 3. Viscosity Reduction versus Additive Concentration for SYNTHOIL Gross Product at 82°C.

To investigate this suggestion, we conducted viscosity measurements on the SYNTHOIL centrifuged product and on the benzene-soluble fraction of the gross uncentrifuged product, with or without Petrowet R added. Analysis of the SYNTHOIL product showed that it contained 1.9 wt % ash, and that the benzene-soluble fraction (obtained by soxhlet extraction) contained 0.5 wt % ash. From the results shown in Tables 6 and 7, we notice that the viscosity for the centrifuged SYNTHOIL product or the benzene-soluble fraction is reduced by about the same magnitude as the viscosity of the SYNTHOIL uncentrifuged product (shown in Table 5). This suggests that the viscosity reduction observed was not due to the effect of additives on suspended solids, but rather to the interaction (or reaction) between the additives and the liquid portion of the SYNTHOIL product.

As reported by Sternberg and coworkers,[1] the viscosity of the SYNTHOIL product is greatly affected by the amount of asphaltenes present (for instance, at 82°C, the viscosity of the product is 175 cp with 30% asphaltenes and 80 cp with 28% asphaltenes). We therefore think that the viscosity reduction is probably due to the interaction (or reaction) between the additives and the asphaltenes, and we are doing experiments to verify this.

Flocculation-Settling Test

We have found that simple conventional tests (capillary-suction test, Buchner-funnel test, etc.) that are used in sewage treatment for flocculant evaluation are not suitable for coal-liquefaction products. We therefore modified a sample analysis technique of Gorin *et al.*[2] to assess the effects of additives for inducing flocculation of SYNTHOIL gross product.

In the modified technique, the additive evaluated is first mixed thoroughly with the product oil in a graduated glass cylinder, which is partly immersed in a constant-temperature bath. A pipet is then immersed a prescribed depth into the oil and small samples are taken at various time intervals for analysis. By comparison of the solids content of the oil samples pipeted at different times, it should be possible to determine the settling rate of the interface between the settling solids and the clear oil phase and, accordingly, to assess the effect of the additives. An alternative to the above procedure is as follows: after the mixture settles for a prescribed time at a certain temperature, it is chilled below the solidification point of the liquid and separated into several portions, and each portion is then analyzed for solid content. This approach also allows the settling rate of the interface to be determined.

We have completed assembly of the testing unit and have started to evaluate the additives for settling of solids in the SYNTHOIL gross product.

Laboratory Precoat Filtration

Precoat filtration is the most common solid-liquid separation technique in use today for coal-liquefaction processes. In principle, the filtration rate is increased when the filtrate viscosity is reduced or when the suspended solid particles flocculate. However, the use of additives may also have side effects on other physical properties[3] (*e.g.*, they may affect surface forces of solid particles) that in turn affect filtration performance. These side effects might either augment or diminish filtration. We, therefore, plan to construct a small precoat filtration unit to further evaluate additives that show promise in viscosity reduction and/or flocculation tests. These studies will also provide information needed for designing a large-scale precoat filtration facility.

Our precoat filtration unit will be similar to that being used by researchers in Oak Ridge National Laboratory.[4] It will consist of a filter tube (3/4-in. ID, 8-in. long) with a support for the precoat, an oil jacket system to maintain the selected filtering temperature, a continuous weighing device; and a pressurizing system. However, to increase the filtration rate and to facilitate the transfer of product oil, the diameter of our filter tube will be larger than that used by Oak Ridge researchers.

Particle-Size Analysis of SYNTHOIL Solids

Knowledge of particle-size distributions of suspended solids in liquids is useful for the design of solid-liquid separation processes. We therefore determined the solid-size distribution of the SYNTHOIL products we have received.

Table 6. Viscosity versus Temperature for Centrifuged SYNTHOIL Product -- with or without Additives

Additive (wt %)	Viscosity (cp)				
	82°C	100°C	120°C	100°C	82°C
0	267	87.2	37.2	87.9	289.
Petrowet R					
0.43	258 (3.3)[a]	84.4 (3.2)	36.0 (3.2)	84.9 (3.4)	277. (4.2)
0.96	234. (12.4)	77.1 (11.6)	35.4 (5.1)	82.5 (6.1)	268. (7.3)

[a] Percentage reduction in viscosity.

Table 7. Viscosity versus Temperature for Benzene-Soluble Fraction of Uncentrifuged SYNTHOIL Product -- with or without Additives

Additive (wt %)	Viscosity (cp)				
	84°C	100°C	120°C	100°C	84°C
0	231	81.3	30.4	81.2	231.5
Petrowet					
0.50	208 (9.9)[a]	73.6 (9.5)	29.2 (3.9)	76.6 (5.7)	218 (5.8)
1.02	196. (15.2)	71.2 (12.4)	28.5 (6.3)	73.5 (9.5)	211. (8.9)

[a] Percentage reduction in viscosity.

Three types of solids were investigated. They are: (1) the benzene-insoluble fraction of the gross product from the SYNTHOIL pilot plant, (2) the ash from the gross product, and (3) the ash from the SYNTHOIL (centrifuged) product. The benzene-insoluble fraction was obtained as residues from solvent extraction, and the ashes were prepared by gradually heating the product samples in a muffle furnace to 700°C. The size distributions of these solids (Fig. 4) were measured by using a model "A" Coulter counter, manufactured by Coulter Electronics, Inc.

For the gross product from SYNTHOIL, about 12 wt % of the ash in the benzene-insoluble fraction has a particle size less than 6 μm, and about 30 wt % of its ash particles are smaller than 6 μm. For the SYNTHOIL product (centrifuged), the percentage for ashes smaller than 6 μm increases to about 50%, as expected. The median particle sizes are 12 μm, 7 μm, and 6 μm for the benzene-insoluble fraction, the ash, and the SYNTHOIL product.

Since high-temperature ashing might alter the form of the ash, the size distribution of the high-temperature ash shown in Fig. 4 may not be representative of ashes present in the product oil. By the use of a low-temperature asher, some ash has been obtained in unaltered form and the size distribution of the low-temperature ashes will be compared with that of the high-temperature ash. Also, visual microscopy and electron microscopy will be used to determine the particle-size distribution and to check the results from a Coulter counter.

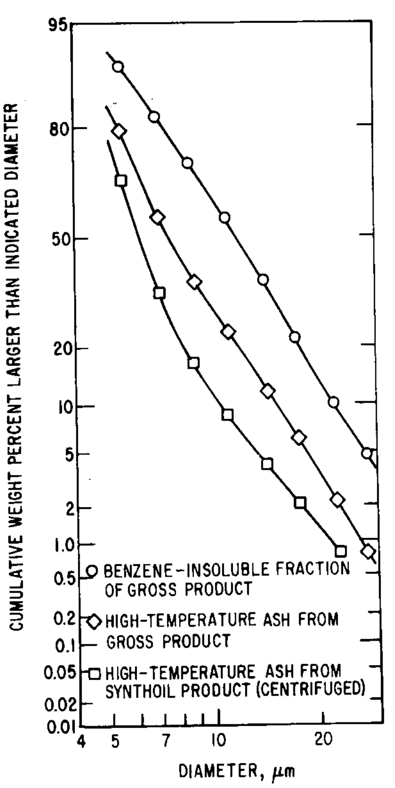


Fig. 4. Particle-Size Distribution of Solids.

IV. CATALYST TESTING
(R. Lo, T. Cannon)

The objective of this task is to evaluate new commercially available catalysts for use in the SYNTHOIL process. Testing will be carried out in a continuous unit at the operating conditions of SYNTHOIL. Results gained from this study will be used to identify the better catalysts. Screening of the catalysts selected will then be done on the long-life test unit at PERC to optimize catalyst selection for use in a 10 ton per day (TPD) SYNTHOIL process development unit currently under design.

As was reported in the preceding quarterly report (ANL-76-2), this work will be performed in two phases.

Phase I. Design, Construction, and Shakedown of Test Unit

We have designed (in consultation with PERC) and will construct a continuous high-pressure coal liquefaction unit wherein catalysts may be tested under SYNTHOIL process conditions, 450°C and 2000-4000 psi. The unit will have the capacity to process at least 4 oz/hr of coal slurry consisting of 35-50 percent coal in recycle oil. The design includes sufficient instrumentation and automation so that it can be operated continuously with minimal attention for periods of no less than 12 days.

Phase II. Tests of Catalysts

We will select, with PERC's agreement, four or more commercially available hydrodesulfurization catlaysts and will test them in the catalyst test unit in 300-hr continuous runs to determine the intermediate life and product variability at 2000 psi and 4000 psi for each catalyst. Product samples will be collected at least once every 24 hr during the run and will be analyzed to determine: coal conversion; sulfur, nitrogen, and oxygen removal; hydrogen consumption; and the viscosity and specific gravity of the gross product oil.

Procurement of Experimental Apparatus

In the previous quarterly report (ANL-76-2), we presented a general description and a schematic flow diagram of the high-pressure Coal Liquefaction Catalyst Test Unit. The test unit is being built by an outside contractor. In order to expedite construction of the unit, components with anticipated long delivery times were ordered. These components will be incorporated into the test unit by the contractor. The function and the current status of all the long-delivery-time components are described below:

Low-Pressure Slurry Circulation and Feed Pump

This pump supplies agitation and mixing to ensure slurry suspension; it also supplies a high suction pressure to the high-pressure slurry pump to prevent loss of pump prime.

A model 3L2-SSF-X Moyno pump was ordered. This is a progressing-cavity type of pump of stainless steel construction; it has a capacity of 50 gal/hr and a 60-psig discharge pressure. This pump has been received.

High-Pressure Slurry Feed Pump

This pump accepts slurry from the low-pressure slurry feed pump loop and injects it at high pressure (up to 4000 psi) into a hydrogen stream to form a gas-slurry mixture for subsequent reaction in the preheater and reactor section.

Two Milton Roy Model MR1-23-49SM Milroyal Simplex pumps, one to serve as a spare, have been ordered. This model is a positive-displacement pump constructed of 316 stainless steel with a 440FM stainless steel plunger; it is rated for 95°C service. It has a capacity of 0.77 gal/hr and a maximum discharge pressure of 4000 psig. These two pumps are scheduled to be received in April.

Primary Gas Compressor

This compressor will be used to increase the pressure of hydrogen (from tanks at regulated pressures of 600 psi or below) to the high pressure of the system.

A Model 46-13421 compressor made by American Instrument Co. has been ordered. This is a diaphragm type compressor of stainless steel construction with a capacity of 20 scfh at 600 psig suction and 4000 psig discharge pressures. This compressor is scheduled to be delivered by April 30, 1976.

Recycle Gas Compressor

This is a compressor which recirculates hydrogen around the loop through the reaction section.

A Model LC-10 Whitey Compressor was ordered. It is a diaphragm compressor of stainless steel construction with a capacity of 120 scfh at 3500 psig inlet and 4500 psig discharge pressures. It has been received.

High-Pressure Wash-Water Pump

This pump will be used to inject water into the effluent gas stream to prevent deposition of ammonia and ammonium salts on the process tubing walls when the gas stream is cooled.

A Model CPES-2 Lapp pump was ordered. It is a combination piston-diaphragm pump of stainless steel construction with a capacity of 0.65 gph at 5000 psi discharge pressure. This pump has been received.

Preheater Furnace

This furnace will be used to heat the gas-slurry mixture in the preheater tube to the reaction temperature before the slurry flow into the reactor.

We have ordered a Model 54472-S Lindberg furnace. It is a vertical split-tube furnace with a single heating zone. It has a 3-in.-ID by 36-in.-long heating chamber and is rated at 6500 W. Maximum operating temperature is 1200°C. THis furnace has been received.

Reactor Furnace

This furnace will be used to maintain the catalyst-packed reactor tube at the desired operating temperature.

A Model 54677 Lindberg furnace was ordered. It is a vertical split-tube furnace with three heating zones. It has a 5-in.-ID by 36-in.-long heating chamber and is rated at 10.4 kW. Maximum operating temperature is 1200°C. This furnace has been received.

High-Pressure Gas-Liquid Separator

The effluent stream from the reactor contains product oil, unreacted coal, ash, and hydrogen. This mixture flows into a separator where the gas phase (95% hydrogen) disengages from the liquid. The liquid product is drained periodically into a low-pressure product receiver, through two throttling valves. The gas effluent from the separator is scrubbed with high-pressure water to remove ammonia and hydrogen sulfide. The gas-water stream is then cooled through a condenser and is flushed into another high-pressure separator where the gas disengages from the water. Water is drained to a low-pressure receiver, while the gas stream is recycled back into the reactor section.

Both separator vessels are designed for use at 5000 psig and 300°C under hydrogen atmosphere. They will have a capacity of 5 gal. The purchase order for the construction of these vessels has been placed with General Atomics Co., San Diego, California. Delivery in mid-June is expected.

High-Pressure Surge/Knockout Pot

Two surge/knockout pots will be required. One will be used to trap the liquid (oil or water) entrained in the recycle hydrogen stream. The other will be used to dampen fluctuation of gas flow discharged from both compressors. These two pots will have a capacity of approximately 1 liter. They are designed for use at 5000 psi and 300°C.

These vessels were also ordered from General Atomics. Delivery in mid-June is expected.

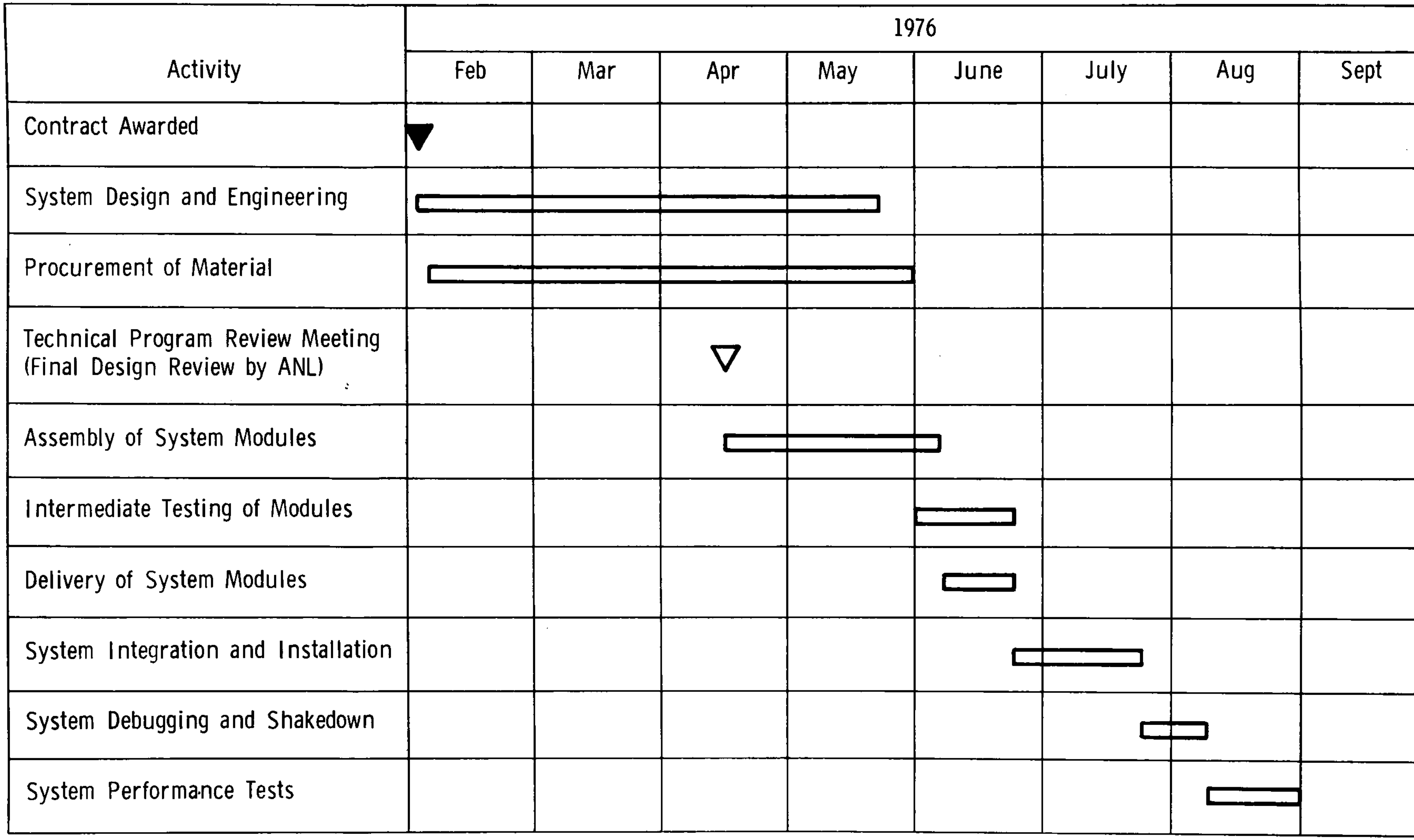
Fabrication of Catalyst Test Unit

Bids for the construction of the Catalyst Test Unit have been received. After a review of all the bids, a contract was awarded to Xytel Corporation, Alsip, Illinois, on February 2, 1976. According to the project schedule (Table 8) submitted by Xytel, fabrication, assembly, and shipment of the five individual modules (slurry delivery module, gas delivery and recirculation module, reactor module, downstream separation and depressurization module, and control console) will be completed by June 30, 1976. It is estimated that installation, debugging, and system performance testing will be completed by the end of August 1976.

Reuse of Product Oil

Due to the limited quantity of recycle oil that will be supplied to us by PERC, most of the effluent product from our experimental runs will have to be

Table 8. Project Schedule for construction of Catalyst Test Unit



clarified and reused. We have selected centrifugation as the method for separating the solid materials, ash, and unreacted coal from the liquid product. A purchase requisition requesting bids for a centrifuge has been submitted.

Facility Preparation

The Catalyst Test Unit and the heat transfer test unit (Task II) will be installed in a building designed for conducting experiments involving large quantities of hydrogen. This building has a high bay experimental area equipped with two blow-out side walls and explosion-proof light fixtures and electrical conduits. The control room is separated from the high bay area by a solid concrete wall. This building had been shut down for approximately three years without heat, electrical power, or other utilities. Work to restore existing building utilities and services and to add three offices for operating personnel is about 70 percent completed.

All slurry transfer lines and slurry holding tanks (feed tank, blowdown pot, product receivers) in the test unit will have to be heated to $\sim$95°C to maintain adequate slurry viscosity. Steam heating will be employed for safety reasons. A high-pressure water boiler which can supply 600 lb/hr of 100-psi steam has been ordered.

A supply of hydrogen will have to be maintained during experiments. The estimated daily hydrogen consumption by the catalyst test program and the heat transfer coefficient test program (Task II) is between 2000 and 4000 scf. We are discussing with Linde Specialty Gases the most suitable and economical means for hydrogen supply and storage.

Feed and Product Analyses

The feed coal bed will be characterized by its proximate and ultimate analysis. The heating value of a coal sample will be measured also. The percentages of the various forms of sulfur compounds (sulfate, pyritic, and organic) in the coal sample will be determined.

The gross product effluent from the reactor (uncentrifuged product oil) will be analyzed for benzene-insoluble, asphaltenes (benzene-soluble, pentane-insoluble), oil (pentane soluble), and ash contents. Elemental analysis will be done to determine the hydrogen, nitrogen, oxygen, and sulfur contents of the product oil. Viscosity, specific gravity, and heating value of the product oil will also be measured. Solvent analyses and elemental analyses of the centrifuged liquid product (recycle oil) and the centrifuge cake will also be performed.

Arrangements have been made with our analytical group to meet the analytical needs described above. A trip to PERC by one member of the analytical group for the purpose of discussing the details of each analytical method is being planned.

V. TRANSPORT PROPERTIES IN MULTIPHASE FLOW*

(K. Javdani,** T. Cannon)

In most coal liquefaction processes including the SYNTHOIL process, a slurry composed of fine coal particles and the recycled product oil flows in a mixture with a pressurized stream of hydrogen. In the SYNTHOIL process, this three-phase mixture is pumped into a preheater where its temperature rises to 450°C. At this temperature and under a hydrogen pressure of 2000-4000 psi, the molecular coal structure is broken up and most of the coal is converted to a liquid that is soluble in the carrier oil. The mixture then enters a fixed-bed catalytic reactor where additional liquefaction of coal particles, molecular cracking, and desulfurization occur. Subsequently, coal ash is removed, producing a clean fuel product.

Optimum scale-up and design and efficient operation of the SYNTHOIL process is dependent on information about the flow characteristics of the three-phase mixture at various stages of the process. Figure 5 shows a schematic representation of one of the stages in the process--namely, the tubular preheater. Heat transfer coefficient data is needed for the design of such heat exchangers. Such data is normally obtained by measuring the inlet and outlet bulk temperatures and the heat flux from the wall. However, the interpretation and conclusions derived from such data depends on the flow condition in the tube. Moreover, utilization of this data in the actual SYNTHOIL process requires that the flow patterns existing in the actual scaled-up heat exchanger be identified.

Unfortunately, in the case of multiphase flow, identification of the flow patterns is not easy. Actually, at different relative velocities of the phases, different types of flow patterns would be established, resulting in different heat transfer coefficients. Figure 6 shows typical flow patterns observed in the upward flow of air-water mixtures in cylindrical tubes.[5] Different flow patterns are established when the velocity of each phase is changed. Unfortunately, no similar information is available for three-phase mixtures, and thus an investigation is required to identify the flow patterns that develop in the preheater shown in Fig. 5. Along with the flow patterns, the investigation should include measurement of other flow characteristics needed in the analysis and design of the process. These include measurements of pressure drop, holdup, and distribution of each phase along the tube. Variations of these characteristics as parameters vary in two-phase flow systems which have been reported in the literature can be used as guidelines in the investigation of the more complex three-phase systems.[5,6]

Another important aspect of analysis relevant to the liquefaction process concerns the transfer of mass between the phases during the flow of gas-solid-liquid mixtures. Figure 7 shows a schematic representation of a possible distribution of phases in the preheater and the

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* Proposed New Task

** Research Associate, Argonne National Laboratory and Assistant Professor of Chemical Engineering, Aryamehr University of Technology, Tehran, Iran.

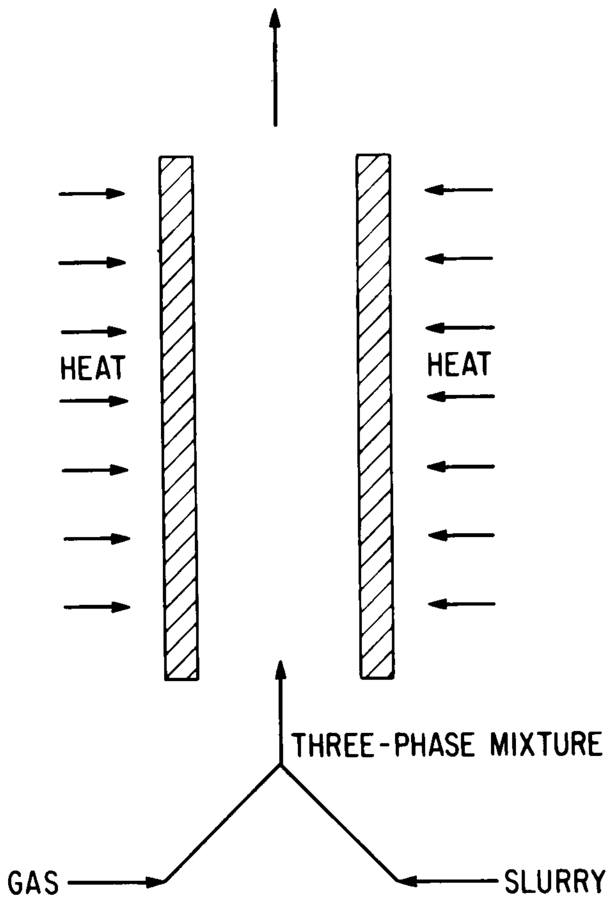

Fig. 5. Schematic Representation of Tubular Preheater.

catalytic reactor. In such a system, a simple model can be proposed to describe the liquefaction of coal particles. According to this model, coal fragments dissolve in the solvent, which is presumably a hydrogen donor liquid. Subsequently, the dehydrogenated solvent is rehydrogenated in the presence of hydrogen. Thus, as seen from Fig. 7, the problem reduces to the determination of solid-liquid and gas-liquid interphase mass transfer rates. Also, it is obvious that the mass transfer coefficient data will depend on the flow patterns and phase configurations, making flow characteristic measurements an essential requirement for the mass transfer work. For this reason, the early stage of the research activities primarily focus on Phase 1 of the program. The schedule of activities is shown in Table 9.

Objectives

The objectives in this experimental work will be to study the flow patterns that develop inside a cylindrical tube during the upward flow of gas-liquid-solid mixtures and to make measurements on the distribution of solid and liquid particles, as well as holdup and pressure drop. This experimental program will produce results

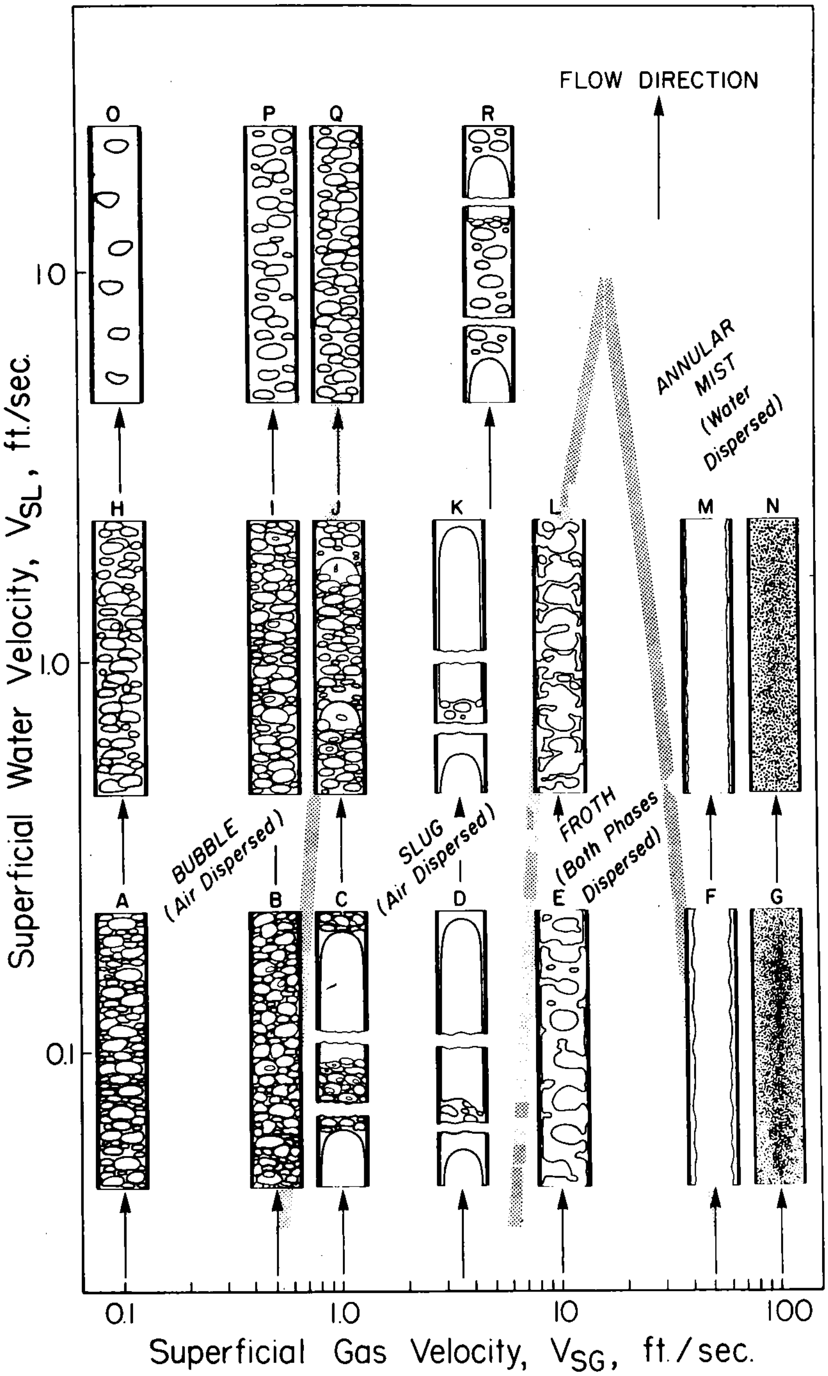

Fig. 6. Flow Patterns Developed in Upward Two-Phase Flow of Air-Water Mixtures in Cylindrical Tubes.[5]

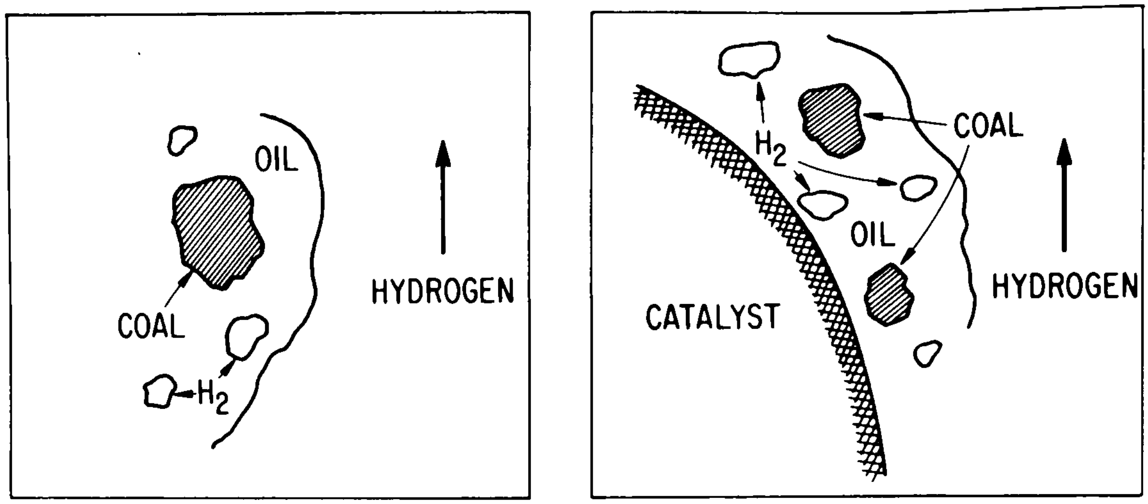

Fig. 7. Schematic Representation of Distribtuion of Phases in (a) preheater, (b) fixed-bed catalytic reactor.

that will be helpful in clarifying certain ambiguities in the flow characteristics encountered in the SYNTHOIL process. Expected results are as follows:

1. The observation and determination of the flow patterns will provide information by which the type of flow existing in the tubular lines of the process under different conditions can be identified.

2. Measurements of particle distribution and holdup will provide a correlation that will predict the quantities of solids, liquid, and gas that exists at any location under different conditions. This information will be especially important in the design of tubular heaters, in which the distribution of particles will affect the heat transfer and mass transfer coefficients and product concentrations.

3. Pressure drop measurements will be used to derive friction factors that are normally used in multiphase flow systems. In addition, abrupt changes in the slope of pressure drop curves will be used in identifying the conditions for transition from one flow pattern to another.

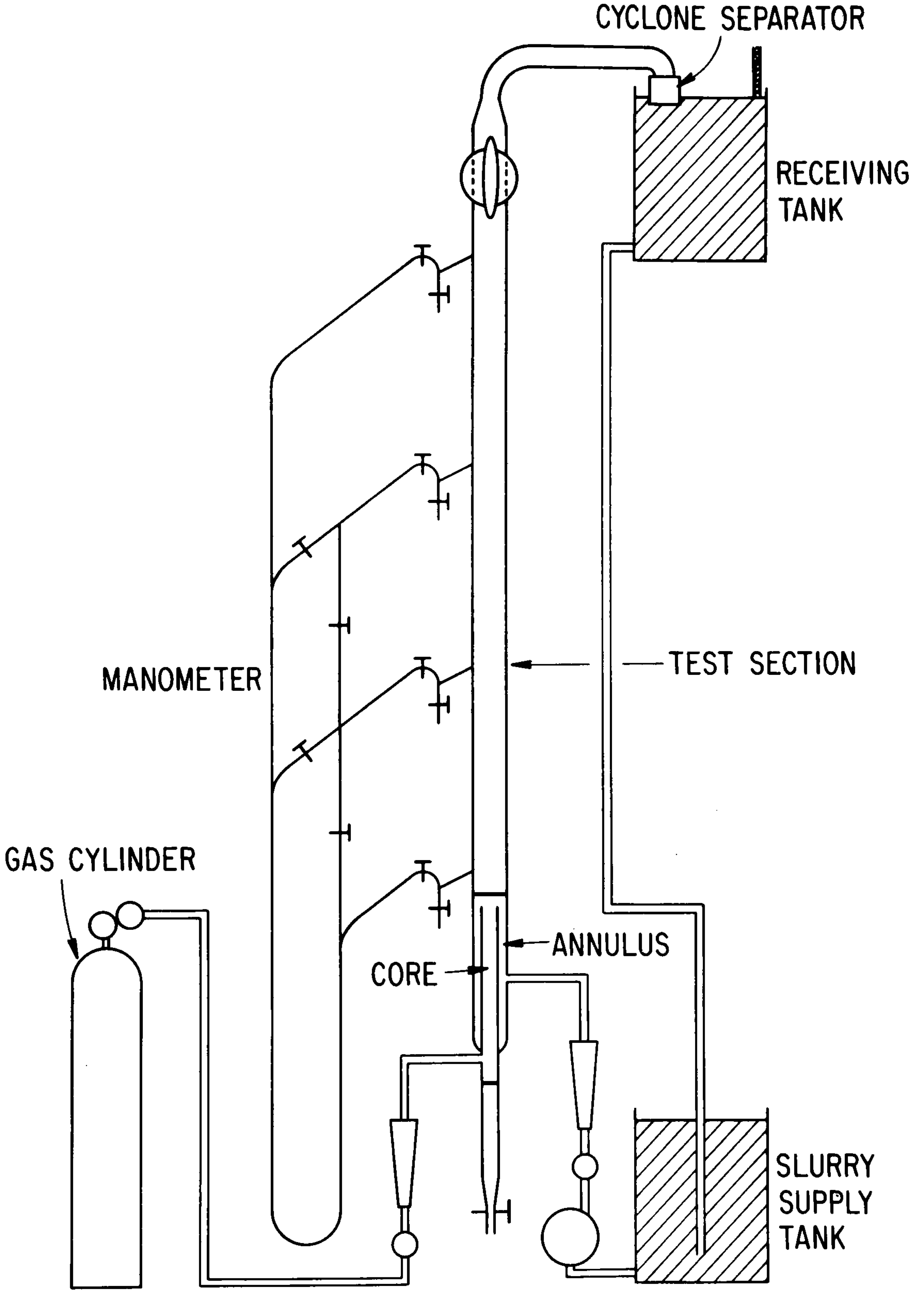

Fig. 8. Experimental Setup (not to scale).

Table 9. Progress Schedule: Transport Properties in Multiphase Flow - Phase 1

Activity	1976											
	Jan	Feb	Mar	Apr	May	June	July	Aug	Sept	Oct	Nov	Dec
Complete the construction and installation of the experimental unit												
Conduct experiments on the characteristics of two-phase flow of mineral oil and helium or nitrogen												
Select suitable solid particles to be used in the three-phase flow experiments												
Study rheological properties of the solid-liquid (slurry) mixture												
Conduct experiments on the characteristics of gas-solid-liquid mixtures												
Start analysis of data and compare with the correlations that exist for two-phase flow												
Design, construct, and install suitable experimental unit for mass transfer experiments												

Experimental Procedure

The experimental unit used for measuring the flow characteristics is shown schematically in Fig. 8. It consists of three test sections (Fig. 8 shows only one of the test sections) made of glass tubes, each 6 ft long, one each with 0.25-, 0.50-, and 1.25-in. inside diameters. The experiments are carried out at room temperature and atmospheric pressure. In this scheme, the slurry is pumped into the annular space of the inlet device, through which it enters the bottom of the test section. Gas, supplied from a pressurized cylinder, enters the test section from the core of the inlet device. After the mixture leaves the test section, it flows through a cyclone separator into a receiving tank. Four pressure taps are provided on the side of the test section at intervals two feet apart; they are connected to a differential manometer for pressure drop measurements. The holdup in the test section is measured by shutting off the slurry and gas lines and simultaneously closing the stopcock on the top of the test section. Thereby, the slurry is captured in the test section and its amount measured.

The establishment of different flow patterns and their transition from one regime to another is detected visually and also by examining the discontinuities in the pressure drop curves. The distribution of phases is determined by high-speed photography of a thin diagonal plane of the flow inside the test section illuminated from outside by a laser beam and rotating mirror.

The effect of the following parameters on the flow characteristics will be examined:

1. Gas and slurry flow rates

2. Physical properties of the fluids

3. Diameter of the test section

4. Concentration of the solid phase in the slurry

5. Size distribution of the solid phase in the slurry

The working fluids have been chosen whose physical properties at ambient conditions are very close to the physical properties of fluids in the SYNTHOIL process at the process conditions. Preliminary experiments on the two-phase flow of white mineral oil (23.6 cp, 0.841 g/cm^3 at 25°C) and helium or nitrogen have indicated that the experimental setup is capable of providing the required data. Experiments on three-phase flow systems will start in the near future.

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