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4-13-77**COAL LIQUEFACTION SUPPORT STUDIES****Task I. Heat of Reaction of Hydrogen
with Coal Slurries****Task II. Heat Transfer Coefficient****Quarterly Report for
October–December 1976****by****J. Fischer, R. Lo, T. Mulcahey,
D. Fredrickson, T. Cannon, and A. Jonke**

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Task II. Heat Transfer Coefficient

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October—December 1976

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J. Fischer, R. Lo, T. Mulcahey
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Chemical Engineering Division

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Previous reports in this series

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COAL LIQUEFACTION SUPPORT STUDIES

by

J. Fischer, R. Lo, T. Mulcahey,
D. Fredrickson, T. Cannon, and A. Jonke

ABSTRACT

This work is in support of the development of processes such as the SYNTHOIL process for converting coal to a liquid fuel that has a low sulfur content and is suitable for use in power production.

In the SYNTHOIL process for converting coal to a low-sulfur fuel oil, coal is liquefied 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/SiO₂-Al₂O₃ catalyst. Turbulent flow of fluid prevents the coal's mineral matter from settling and plugging the reactor. In a 1/2 ton per day pilot plant at PERC, the gross liquid product is centrifuged to remove unreacted solids, producing a low-sulfur, low-ash liquid fuel.

The work for the remaining portions of this program includes tasks I and II, started in September 1975.

Task I, Heat of Reaction of Hydrogen with Coal Slurries

The heat of reaction of coal-oil slurries with hydrogen will be determined in the presence and absence of catalysts at 2,000-4,000 psi and 400-475°C, using a calorimeter. Calorimeter construction is complete, and performance evaluation and calibration have been started.

Task II, Heat Transfer Coefficient

The coefficients of heat transfer from SYNTHOIL reactor fluids to heat exchangers will be determined in the following ranges of conditions: pressures of 2,000-4,000 psi and temperatures to 465°C. The test unit has been installed. Acceptance testing will be done during January 1977.

SUMMARY

Task I, Heat of Reaction of Hydrogen with Coal Slurries

A calorimeter has been built by Calorimetrix Inc. of Boulder, Colorado, to be used to obtain heat of reaction data for the hydrogenation of coal slurries typical of those found in the reactant stream of the SYNTHOIL process. Calibration tests are now being performed in Boulder prior to shipment of the calorimeter to Argonne in January 1977.

Task II, Heat Transfer Coefficient

An experimental unit, which will be used to obtain measured coefficients of heat transfer from both SYNTHOIL coal slurry process feed and product stream to container walls, for use in future design of heat transfer equipment for the process, is near completion. The test unit will have feed capacity of slurry and hydrogen flow rates comparable to those in a plant designed for 1/2 ton/day capacity. All individual modules for the test unit have been delivered to the facility at Argonne and interconnection of the various modules has been completed by the contractor.

Fabrication of the heater and cooler components of the heat transfer test section is complete, and assembly of the components to form the test section module is almost complete. Preliminary testing and calibration of the components were done prior to assembly.

PROJECT PLAN AND STATUS

The project plans are depicted in Tables 1 and 2 for the two active tasks.

Task I, Heat of Reaction of Hydrogen with Coal Slurries

At the end of this report period (December 31, 1976), final testing of the calorimeter was being done by the vendor before shipment of the test unit to Argonne in January.

Task II, Heat Transfer Coefficient

At the end of this report period (December 31, 1976), installation of the test unit was 90% complete. Completion of the test facility is expected to be accomplished before the end of January 1977.

Both tasks are scheduled for completion in September 1977. A final report will be completed by the end of that month.

Table 1. Task 1. Heat of Reaction of Hydrogen with Coal Slurries, Work Plan FY-1977

	1976				1977							
	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUNE	JULY	AUG	SEPT
Heat of Reaction Calorimeter	Design and Construction Contract Awarded February 1976											
Fabrication ^a												
System Testing												
Ship and Reassemble												
Heat of Reaction Determinations												
Performance Testing												
Establish Experimental Procedure												
Hydrogen & SYNTHOIL												
2000 psi 450°C												
Hydrogen and Coal Slurry												
2000 psi 450°C												
H ₂ + SYNTHOIL and Catalyst												
2000 psi 450°C												
H ₂ + Coal Slurry + Catalyst												
2000 psi 450°C												
Hydrogen + SYNTHOIL												
4000 psi 450°C												
Hydrogen + Coal Slurry												
4000 psi 450°C												
H ₂ + SYNTHOIL + Catalyst												
4000 psi 450°C												
H ₂ + Coal Slurry + Catalyst												
4000 psi 450°C												
Final Report												
^a Construction of test unit started May 1976.												

Table 2. Task II, Heat Transfer Coefficient, Work Plan, FY-1977

	1976			1977								
	OCT	NOV	DEC	JAN	FEB	MAR	APRIL	MAY	JUNE	JULY	AUG	SEPT
Heat Transfer Test Unit	Design and Construction Contract Awarded March 1976											
Fabrication, ^a Installation												
Installation Complete												
Acceptance Tests												
Test Module												
Fabrication												
Installation Complete												
Instrument Tests												
Complete Calibrations												
Heat Transfer Coefficient												
Determinations												
Gas Flow												
Liquid-Gas Flow												
SYNTHOIL, Effluent Flow												
Slurry-Gas Flow												
Coal A												
Coal B												
Coal C												
Final Report												
^a Construction of test unit started June 1976.												

TASK I. HEAT OF REACTION OF HYDROGEN WITH COAL SLURRIES
(D. Fredrickson)

A calorimetric system, described previously in ANL 76-117, has been built by Calorimetrics, Inc. of Boulder, Colorado, for determination of the heat of reaction of hydrogen with coal slurries typical of those used in coal liquefaction processes. The basic components of the system are a drop tube, a heated pressure vessel (in which the reaction takes place), and a calorimeter surrounding the pressure vessel.

Calibration tests are being conducted at Calorimetrics in order to meet the acceptance criteria specified in the contract. These include electrical calibrations with a precision (standard deviation) of 1% or better as determined by replicate electrical calibrations. These have been completed successfully. A second calibration test requires that the calorimeter be capable of an accuracy of 1% in a determination of the enthalpy of synthetic sapphire. The latter is near completion, and delivery of the heat of reaction calorimeter is scheduled for the end of January 1977. A packaging plan has been received from Calorimetrics for shipping of the calorimeter. The plan is to ship the apparatus directly to ANL in a panel truck driven by Calorimetrics personnel.

The calorimeter will be installed in a laboratory in the Chemical Engineering Division building. This laboratory has a separate shielded area for the calorimeter.

To accomplish the goal of this task, we need (1) to know the energy released during the reaction and (2) to determine the extent of the reaction. The energy release will be measured in the calorimeter. Extent of reaction (hydrogen consumption) will be obtained from the hydrogen balance between the reactants and products. Upon completion of an experiment, liquid and gas products must be analyzed. Liquid products will be analyzed by conventional means. We are working on procedures for handling and analyzing the gaseous products.

Auxiliary equipment has been set up to determine the optimum conditions for collecting gases in cold traps after a reaction in the calorimeter has been completed. The equipment includes a 2-liter autoclave, with a vessel identical to that used in the calorimeter and a gas-handling manifold. A series of experiments are under way to develop procedures for quantitative determination of hydrogen in the reaction products. The determination of hydrogen in the gaseous products is difficult because hydrogen gas must be separated from the other gases. Most of the gaseous components can be trapped using liquid nitrogen as a coolant. The separation of hydrogen from methane is particularly difficult since methane has a partial pressure of about 1.0 kPa (10 Torr) at liquid N₂ temperature.

In a preliminary test, the autoclave was charged to 6.9 MPa (1000 psi) with a He-5.38% CH₄ mixture and slowly discharged through the two liquid nitrogen traps to condense CH₄. The traps were then warmed, and the collected methane was passed over hot CuO, where it was converted to H₂O. The H₂O was collected in a U tube containing magnesium perchlorate and weighed. We are developing this method to be used for the H₂-CH₄ mixture expected in a calorimeter experiment. At present, the limiting factor in this procedure is the vapor

pressure of CH_4 at liquid nitrogen temperatures, around 1.0 kPa (10 Torr). We are evaluating molecular sieves as a method for collecting the portion of the CH_4 which is not condensed at liquid nitrogen temperature.

TASK II. HEAT TRANSFER COEFFICIENT
(T. P. Mulcahey, R. Lo, T. Cannon)

The objectives of this task are to determine heat transfer coefficients suitable for the design of process feed heat exchangers and product effluent heat exchangers used in coal liquefaction processes and to identify mechanisms for the heat transfer process. Heat transfer film coefficients for mixtures of gas, liquids, and solids in a turbulent flow will be measured for (a) reactor effluent cooled by a stainless steel surface and (b) feed heated by a stainless steel surface. The experimental work will be carried out in a test module in a nominal 1/2-ton-per-day Heat Transfer Test Unit, which will include a slurry makeup system; slurry feed system; heaters; coolers; pressure letdown tanks; and power, flowrate, and temperature instrumentation.

The following ranges of conditions will be covered:

Pressure:	2,000-4,000 psig (14-28 MPa)
Temperature	200-465°C

Three different coals suitable for liquefaction will be studied in the investigation. Slurries will be used having the compositions of SYNTHOIL gross product (unfiltered oil) and SYNTHOIL feed containing 30% by weight of finely ground coal.

A. Heat Transfer Test Unit*

All modules and the unit control console have been delivered to us from the commercial firm that is building the test unit. Integration of the various modules is completed. Instrumentation and power wiring will be completed by January 15, 1977.

*Described in detail in ANL 76-117. The basic operation of the unit is as follows: A coal slurry consisting of an oil with physical properties similar to those of SYNTHOIL and a 200-mesh coal is mixed and heated in the 140-gal slurry make-up tank. A mixer and a rotating cavity recirculation pump maintain the slurry in suspension; the pump also provides the pressure to transfer the slurry to the 10-gal slurry feed tank. A mixer and recirculation pump connected to the slurry feed tank maintain slurry suspension, and the pump provides slurry at pressure to the suction side of the piston-type high-pressure pump. The high-pressure pump supplies the slurry to the entrance of the test module at 4,000 psi.

Hydrogen supplied to the unit at 500 to 1,000 psi, is compressed to 4,000 psi and merged with a recirculating hydrogen stream. The recirculating hydrogen is merged with the slurry before it enters the test section. The gas/slurry mixture passes into the heaters where a heat transfer coefficient is measured; the mixture then passes into the coolers where another heat transfer coefficient is measured.

The gas and liquid solid phases are separated in the high-pressure receiver. The gas stream is washed and cooled and returned via another gas compressor as the recirculating hydrogen stream. The liquid SYNTHOIL type effluent and water wash are collected in low-pressure receivers and periodically sampled and removed from the system.

In order to protect other equipment and personnel in the building during operation, interior steel walls have been erected around apparatus containing high-pressure gas.

Blast mats are to be placed along the exterior blow-out walls of the building to prevent any debris or flying pieces from being shot through the wall in case of an explosion. The exterior walls will be covered by matting up to the 10-ft level except where the high-pressure receivers extend the needed coverage up to the 15-ft level.

B. Test Module

Fabrication of the test module, which contains the heating and cooling test components, is almost complete. All parts for the heaters, mixers, and coolers have been manufactured by the ANL shops or procured, and most of the individual heaters, mixers, and coolers have been fabricated. Assembly of the heaters into one leg of the L-shaped test module has been initiated. Fabrication of the control and instrumentation racks has been initiated and the shunts to be used for power calibration of the individual heaters have been mounted. Testing and electrical calibration of the test module and data acquisition systems were initiated, using a dummy test section heater. The blower that supplies compressed air to the coolers was delivered, and installation of the blower and compressed air piping is completed. Installation of the air flowmeters and piping connection for the test section coolers will be completed by January 15, 1977.

Figure 1 is a photograph of a SYNTHOIL mixer lying on its side. The slurry-gas mixture will enter the left end and be mixed, the bulk temperature will be measured by a thermocouple inserted into the thermal well at the top near the right-hand end, and the mixture will exit at the right end. Figure 2 is a photograph of a partially assembled helix cooler; the five wires at each end are thermocouples which are attached to the outside wall of the 3/8-in. stainless tubing containing the slurry-gas mixture. Eight of the thermocouple wires are embedded in grooves of the inner flow surface of the tube which is surrounded by the helix. The thermocouples are held in place by a high-temperature chlorine-free epoxy as shown in Fig. 3, which shows the cooler prior to helix installation.

Figure 4 is a photograph of the internal assembly of the straight tube cooler; eight thermocouples are attached to the 3/8-in. tube for monitoring the external wall temperature. Figure 5 is a photograph of one of the six heaters prior to assembly into the test section and prior to installation of any thermocouples.

Fig. 1. SYNTHOIL Mixer Lying on Its Side

Fig. 2. Helix Cooler, Partially Assembled

Fig. 3. Helix Cooler. Partial Assembly Prior to Helix Installation

Fig. 4. Thermocouple Assembly on Straight Tube Cooler

Fig. 5. Heater

C. Test Program

The Heat Transfer Coefficient Measurement Test Unit SYNTHOIL testing program is divided into five main phases as described below.

1. Test Module Characterization Tests

Objective: These tests are designed to obtain calibration and heat loss data for the test module components so that compensation can be made for instrument errors and heat losses in the calculation of heat transfer rates.

Program

a. Calibration will be performed on the following:

- (1) electrical shunts used to measure the current to the test module heaters
- (2) thermocouples mounted on the mixers, heaters, and coolers
- (3) high-pressure liquid pump flow
- (4) air flow monitors for the cooling air supply
- (5) data logging system
- (6) hydrogen recirculation and makeup flow monitor

b. Heat loss determination

The heaters, mixers, coolers, and piping will have heat losses associated with the transmission of heat through the insulation. The values of the heat losses of the various components must be determined to assess their significance and to allow correction, if necessary, for these losses.

2. Tests of Gas Heat Transfer Coefficients at Base Conditions

Objective: Heat transfer coefficients for hydrogen, helium, and nitrogen will be determined for the "clean" system. These coefficients will serve as a base to which the heat transfer for SYNTHOIL fluid can later be compared and as a base for monitoring the fouling of the heat transfer surface during the remainder of the test program.

Program: Heat transfer coefficients of the pure gases will be run on the initial clean system and periodically after each major step throughout the course of the test program.

3. Liquid/Gas Flow Characteristics at Base Conditions

Objective: These tests are designed to provide information about the physical nature of the flow (e.g., slug, annular) as a function of the expected flow parameter variations.

Program: Fluids of different viscosities will be circulated through the system under different gas flow rates to ascertain characteristics associated with different flow regimes.¹

4. SYNTHOIL Effluent Tests

Objective: These tests are to determine the heat transfer coefficients of the SYNTHOIL effluent.

Program: The heat transfer coefficient will be determined in the following range of parameters.

a. pressure: 2,000-4,000 psig

b. temperature: 200-465°C

5. SYNTHOIL Slurry Feed Tests

Objective: These tests are to determine the heat transfer coefficients of the slurry feed material, using three different coals.

Program: The heat transfer coefficients will be determined over the range of parameters listed in 4. above.

REFERENCE

1. K. Javdani, S. Schwalbe, and J. Fischer, "Multiphase Flow of Gas-Liquid and Gas-Coal Slurry Mixtures in Vertical Tubes," Argonne National Laboratory, January 1977, ANL 76-116.

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