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COAL LIQUEFACTION SUPPORT STUDIES

**Task I. Heat of Reaction of Hydrogen
with Coal Slurries**

Task II. Heat Transfer Coefficient

**Quarterly Report for
January—March 1977**

MASTER

by

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



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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

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16. Abstracts A development program is being carried out to obtain information applicable to the Synthoil process for converting coal to liquid fuel of low sulfur content. This report presents information on (1) a calorimeter to measure heat of reaction of hydrogen with coal slurries and (2) the construction status and a test program for the apparatus for measuring heat transfer coefficients of Synthoil feed and effluents:			
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Molybdenum Sulfides		Slurries	
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COAL LIQUEFACTION SUPPORT STUDIES

Task I. Heat of Reaction of Hydrogen with Coal Slurries
Task II. Heat Transfer Coefficient

Quarterly Report for
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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 consists of 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. The calorimeter has been delivered to Argonne and performance evaluation and calibration tests are under way.

Task II. Heat Transfer Coefficient

The coefficients of heat transfer from Synthoil reactor fluids to heat exchanger surfaces will be determined in the following ranges of conditions: pressures of 14-28 MPa, and temperatures of 200-465°C. Installation of the test unit and the test module has been completed. Heat transfer coefficient measurements will be started in early June 1977.

* Some work is report that was performed during April and May, 1977.

SUMMARY

Task I. Heat of Reaction of Hydrogen with Coal Slurries

A calorimeter has been built by Calorimetrics, 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 preheaters and hydrogenation reactors of coal liquefaction processes. This calorimeter is unique in design and incorporates a heavy pressure vessel with a delicate calorimeter. The calorimeter was delivered to Argonne late in January 1977. Since that time, numerous changes have been necessary both to the calorimeter body and the associated electronics.

It was necessary to rebuild the thermopile, which is the main sensing element for control of the calorimeter. This involved some redesign, machining of parts, and reassembly at Boulder, Colorado, by the vendor. Reassembly and further shakedown testing will take place during the second half of May; calorimetric calibrations are scheduled to begin the first week of June.

Task II. Heat Transfer Coefficient

An experimental unit has been completed which will be used to obtain measured coefficients of heat transfer between (1) Synthoil coal slurry process feed streams and container walls and (2) product streams and container walls. The heat transfer data will be used in future design of heat transfer equipment for the process. Installation of the test module, which includes the heating and cooling elements, has also been completed. Preliminary testing and calibration of instruments and components has started. Heat transfer coefficient measurements with gas flow alone will begin in June 1977.

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

Numerous changes have been made to the calorimetric system since delivery in late January 1977. Performance testing is continuing and will be followed by work to determine experimental procedures and heats of reaction of combinations of coal slurry, Synthoil, and catalysts (as outlined in the project plan in Table 1). This project will be concluded in September 1977.

Task II. Heat Transfer Coefficient

A. Heat Transfer Test Unit

Installation of the Heat Transfer Test Unit is complete. High-pressure steam supply lines, steam tracing on process tubings and vessels, and condensate return lines have all been hooked up. All system exhaust gas vent lines have been piped out of the high bay experimental area and brought up above the roof ledge line. The entire test unit has been leak-tested and pressure-tested with nitrogen at 4,000 psig (28 MPa). The functions of all

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

	1977								
	Jan.	Feb.	March	Apr.	May	June	July	Aug.	Sept.
Heat of Reaction Calorimeter	Design and Construction Contract Awarded February 1976								
Design, Fabrication, ^a	■								
Ship and Reassemble		■							
Heat of Reaction Determinations									
Performance Testing		■				□			
Establish Experimental Procedure						□			
Hydrogen and 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								□	
Prepare Final Report									□

^a Construction of test unit started May 1976.

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

	1976			1977								
	Oct.	Nov.	Dec.	Jan.	Feb.	March	Apr.	May	June	July	Aug.	Sept.
Heat Transfer Unit	Design and Construction Contract Awarded March 1976											
Fabrication, Installation ^a	[Solid black bar from Oct 1976 to May 1977]											
Installation Complete								▼				
Acceptance Tests								[Solid black bar]				
Test Module												
Fabrication	[Solid black bar from Oct 1976 to May 1977]											
Installation Complete								▼				
Instrument Tests								[Open bar]				
Complete Calibrations									▼			
Heat Transfer Coefficient												
Determination												
Gas Flow									[Open bar]			
Liquid, Gas Flow										[Open bar]		
Synthoil Effluent Flow											[Open bar]	
Coal-Slurry, Gas Flow												[Open bar]
Prepare Final Report												[Open bar]

^a Construction of Test Unit started June 1976.

safety devices (pressure switch, relief valve) and monitoring devices (pressure transducer, weighing scale) were tested and calibrated at full system pressure, using helium and deionized water as process fluids. Still to be done by the outside contractor who built this unit are performance of a final test with hydrogen at full system pressure (28 MPa) and handing over to ANL all documentation, to include operating and service manuals and parts lists.

A request has been submitted to the ANL shop that a perforated 1-in. steel pipe be laid underground from the location where the hydrogen tube-trailer will be parked to the vicinity of the building exterior wall. The pipe contains and protects the 1/4-in. stainless steel hydrogen feed line which runs from the hydrogen supply tube trailer to the gas intake manifold for the test unit.

B. Test Module

Assembly and installation of the test module, which includes the heating and cooling test components as well as mixers,² is completed. Thermocouples have been installed along the exterior wall of each heating and cooling test element. Some thermocouples were also placed on the transfer pipe between test elements, on the electrodes of the heaters, and inside the insulation material on the piping. All thermocouples except those in the insulation were installed by discharge welding of the individual chromel and Alumel wires directly on the metal surface. As a result, the thermocouples should read the true skin temperature. The entire test module, including test elements, transfer pipes, and cooling air exhaust pipes, was covered with three layers of insulation having a total thickness of 5 in.

Installation of the cooling air flow meters and piping connections for the test section coolers was also completed during the report period.

Installation of electrical power cables from the power supply to each heater electrode is complete. Hookup of thermocouple, voltage, and air flow transducer input channels into the data acquisition system is 90% complete. This program will be concluded in September 1977.

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

The calorimetric system for determination of the heat of reaction of hydrogen with coal slurries typical of those used in coal liquefaction processes has been built but has required additional work to make it operable. The system was described previously in ANL 76-117. The basic components of the system are a drop tube, a pressure vessel (in which the reaction takes place), and a calorimeter surrounding the pressure vessel.

A schematic view of the calorimetric system is shown in Fig. 1. The apparatus is shown in Fig. 2. Half of the aluminum upper radiation shields are removed in Fig. 3 to 5 to show progressively more of the inside. The jacket lid is seen in Fig. 3 and the calorimeter lid in Fig 4. Finally, in Fig. 5 the innermost part is visible--the top of the pressure vessel.

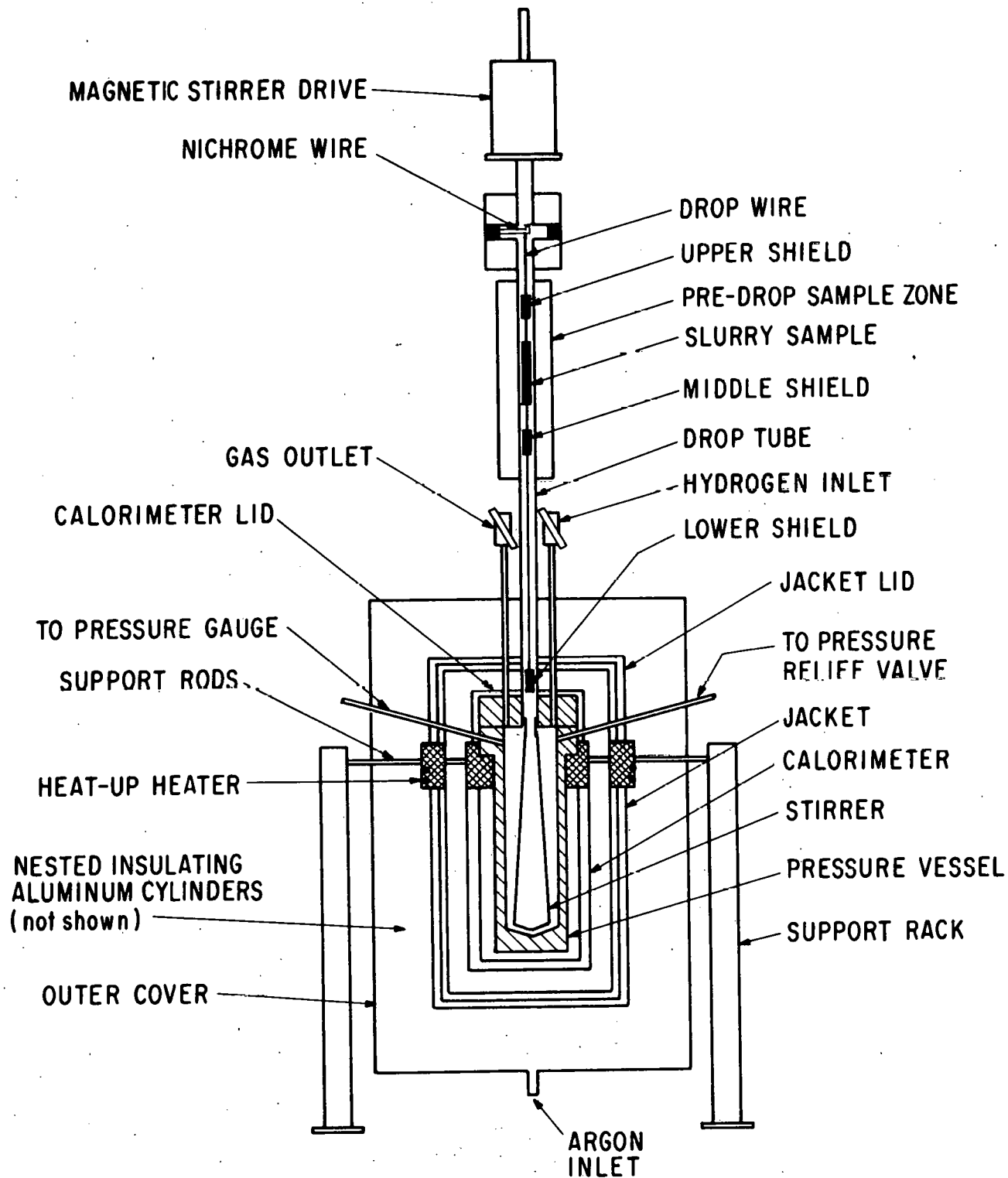


Fig. 1. Heat-of Reaction Calorimeter

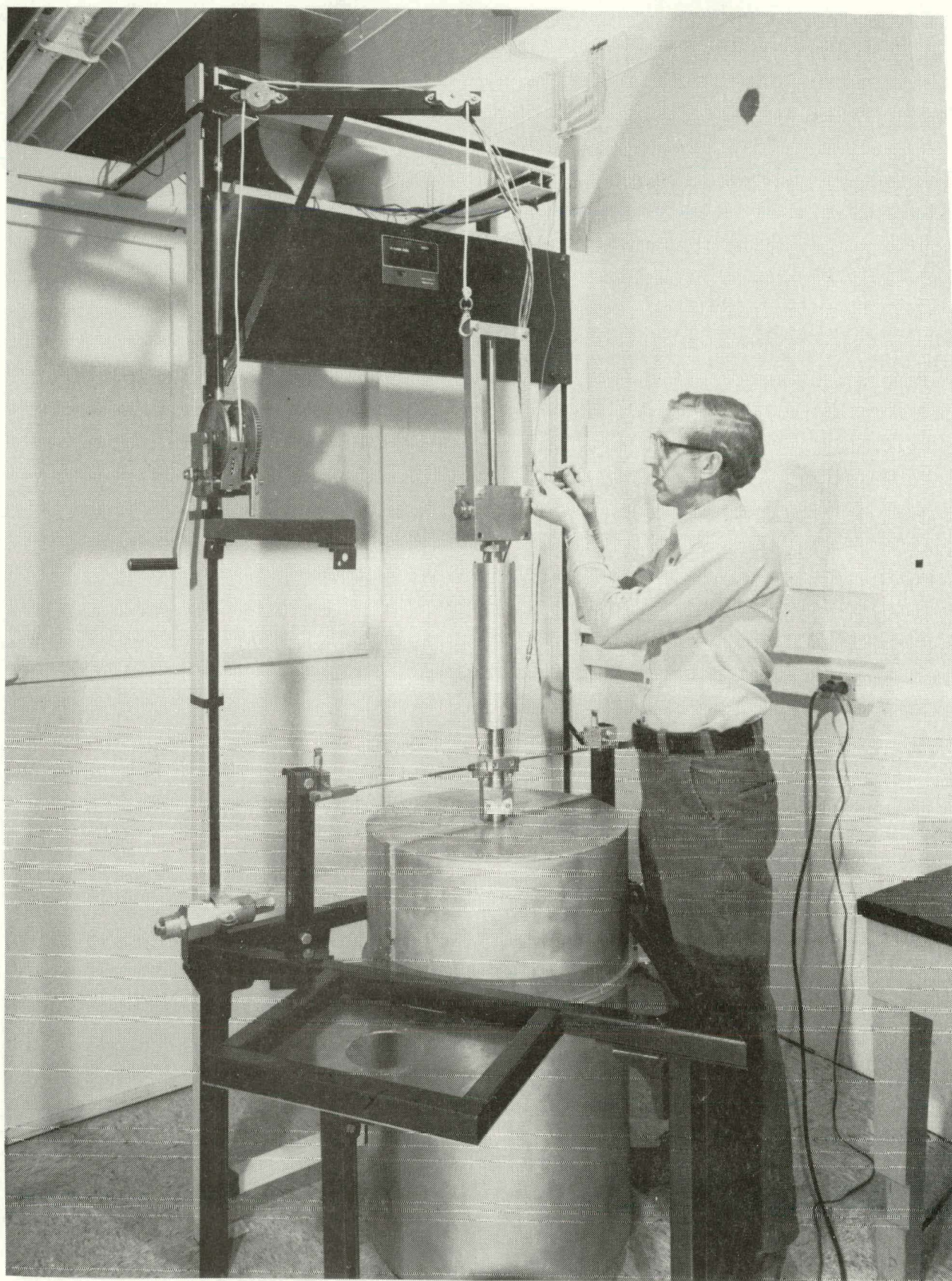


Fig. 2. Calorimeter Assembly

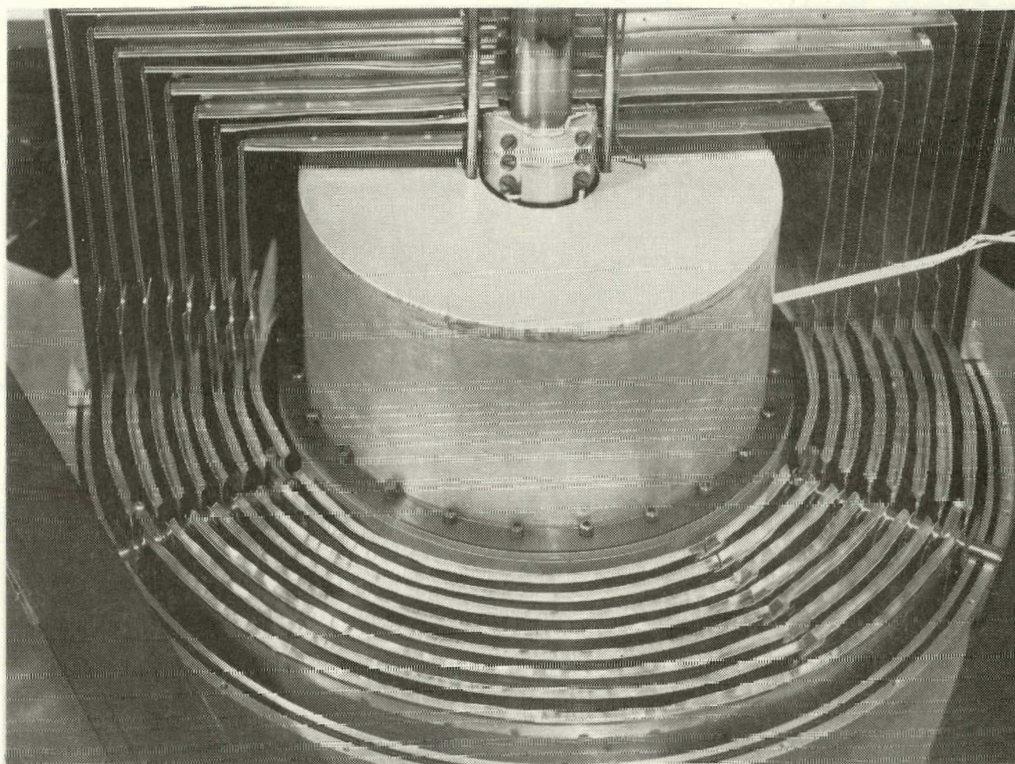


Fig. 3. Top View of Calorimeter Assembly--Jacket Lid, Radiation Shields, and Lower Part of the Drop Tube

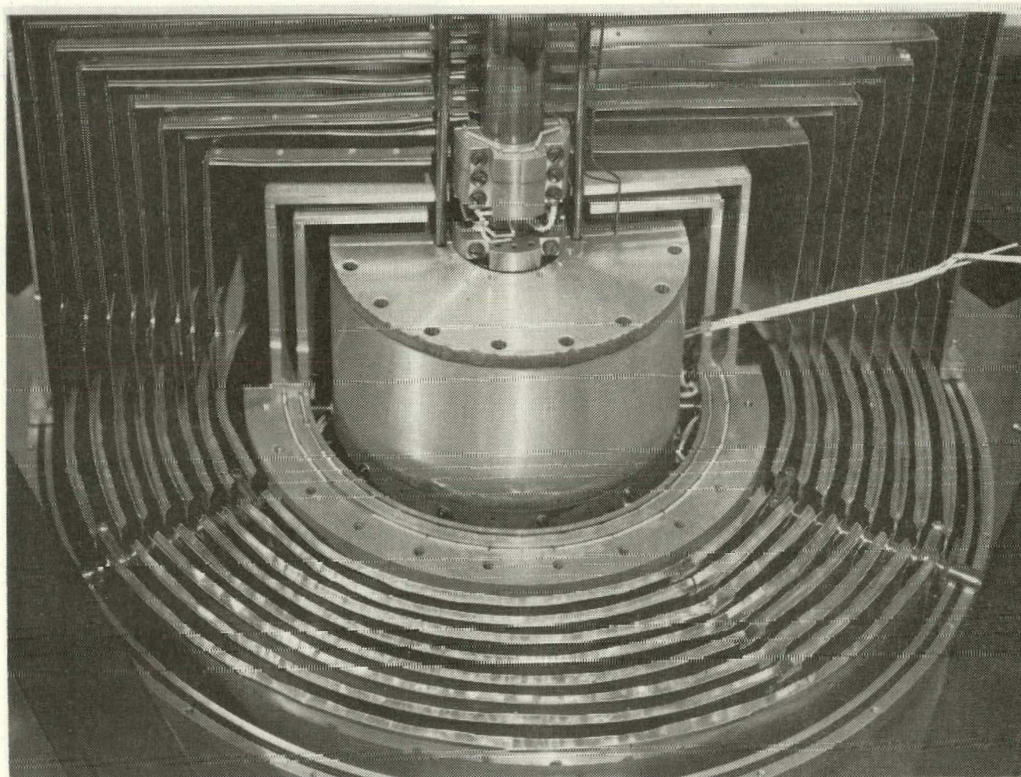


Fig. 4. Top View of Calorimeter Assembly--Calorimeter Lid

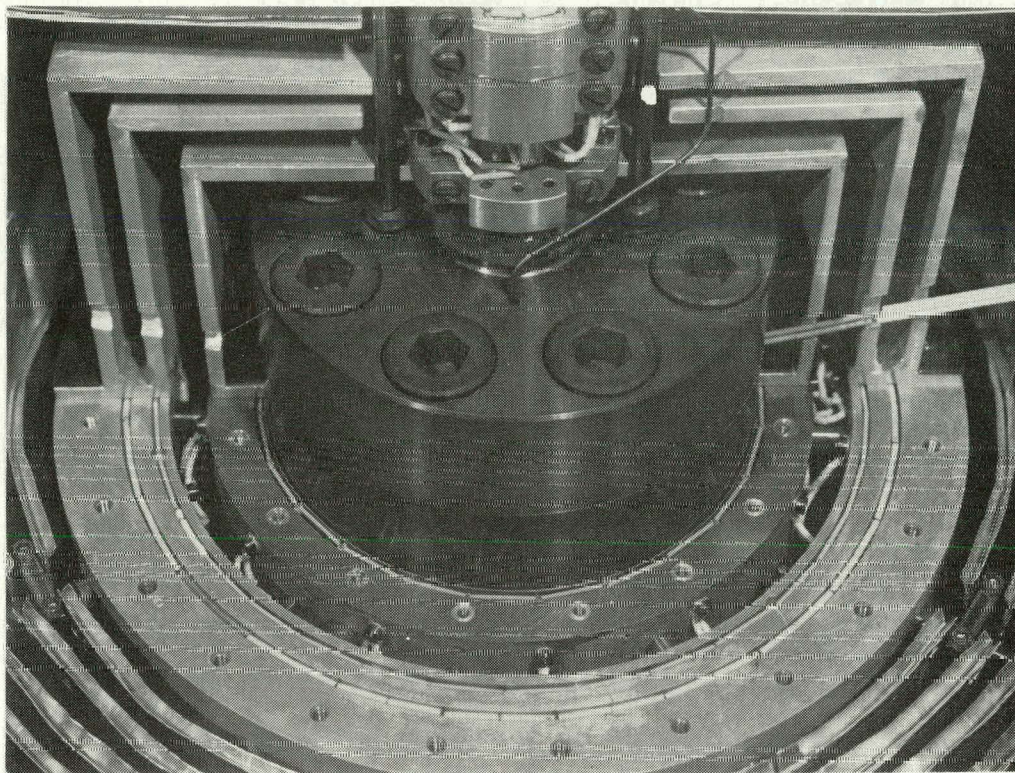


Fig. 5. Top View of Calorimeter Assembly--Top of Pressure Vessel

The calorimeter was delivered to Argonne in late January 1977, via rented truck operated by Calorimetrics personnel. Despite very careful handling during shipment, the heater and thermopile leads became twisted and all had to be replaced. Reassembly by Calorimetrics personnel took a considerable amount of time, and various other mechanical parts had to be worked on also. After reassembly, testing of the calorimeter was incomplete due to malfunctioning of the logic circuitry, making it necessary to return it to the vendor. This unit was returned to Argonne a month later.

There was a series of delays with the electronics external to the calorimeter and then with heaters, thermopiles, and wiring inside the calorimeter. The logic circuitry was returned to the vendor in Boulder twice, and the drop tube power supply was returned once for repair and modification. The vendor has been to Argonne on four different occasions since initial delivery in an effort to get the system into operation.

As of May 1 it appears that the electronic circuitry external to the calorimeter is working. There has been shorting to the ground in the temperature-maintenance heater when the calorimeter temperature reached 460°C. Shorting to the ground, also a problem in the thermopile, occurred because of corrosion of leads, tie downs, and thermocouple junction points. This resulted from the use of MoS₂ as lubricant on the pressure vessel bolts. When heated to 460°C the MoS₂ oxidized to form SO₂, which resulted in corrosion and electrical shorts.

To eliminate the corrosion, MoS_2 will not be used to lubricate the autoclave bolts but another suitable lubricant will be found. The contractor has rebuilt the thermopile; this required some redesign. Synthetic mica was used as an insulator for the original thermopile. The mica was very thin and fragile and difficult to handle. Therefore, we switched to a machinable glass ceramic (Corning). This, along with a centering bushing, allowed simpler installation.

Leaks were found in the pressure vessel closure flange. Personnel of the ANL machine shop assisted by lapping the sealing surfaces. A spanner wrench was used to hold the flange of the pressure vessel during opening and closure; a torque wrench was used to seal the closure. This prevented the applied force from turning the vessel, which force would in turn be transmitted to the delicate electrical leads of the calorimeter heaters and thermopile. The pressure vessel was made leak-free by using proper torquing procedures with adequate lubrication of the bolts.

The work is about 3 months behind the overall experimental schedule. Table 1 is a revised work plan.

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

The objectives of this task are to determine heat transfer coefficients needed for the design of feed heat exchangers and effluent heat exchangers used in coal liquefaction processes and to identify mechanisms of the heat transfer process. Heat transfer film coefficients for mixtures of gas, liquids, and solids in 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,¹ which includes a slurry makeup system; slurry feed system; heaters; coolers; let-down 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

flow of fluids: turbulent

Three different coals suitable for liquefaction will be studied in the investigation. Slurries will be used having the compositions of (1) Synthoil gross product (unfiltered oil) and (2) Synthoil feed containing up to 30% by weight of finely ground coal.

A. Testing Program

1. Electrical Power for Heater Element

Electrical power is to be supplied to the heaters as shown in Fig. 6. The power supplies are d-c. Constant Potential Welders capable of outputting 600 A with voltages up to 50 V. As can be seen in that figure, three power supplies are used to supply the six heaters* with power; each welder supplies two heaters. Commercially available ($\pm 2\%$) current shunts have been mounted in a control chassis (with forced air cooling) to monitor the currents to the heaters. The shunts monitor the total current from the power supply as well as the individual heater currents. By calibration and by forced-air cooling of the shunts, it is expected that the commercial shunts can be used to measure current within 0.1% accuracy.

To prevent current from passing through portions of the test module other than the heaters, the voltage applied to each heater element in a pair must be the same. During construction, the electrical cable lengths to the pairs of heaters were matched to ensure minimum voltage variations to the heaters. Electrical grounding of the heaters at the electrode connections closest to the slurry inlet and outlet of the heating section ensures that no current flows in the process development unit exterior to the section containing the heating elements; each heater element has one ground in order to eliminate floating voltage variation within the heating section.

B. Calibration Testing

Accurate measurement of the current to the test module heaters is accomplished with commercially available current shunts (Table 3). The shunts used are resistors designed to produce 50 mV at their full current rating. They are made of material having almost no resistance variation with temperature. Two parameters are to be verified during calibration of the shunts. The first verifies that the shunts are not temperature-sensitive as a result of design or construction methods; the second is a calibration to determine the accurate value of the shunt resistance under the conditions of normal operation. While calibrating the shunts, the heater elements were disconnected and a water-cooled prototype test heater element was used that had been previously used for practice and personnel qualification for thermocouple installation. Calibration was performed with currents of 87 to 300 A.

Two Leeds and Northrup precision ($\pm 0.04\%$) 300-A shunts (Model 4363) were wired in series with the commercial shunts built into the Test Unit control frame. The first Leeds and Northrup was obtained from the Chemical Engineering Division Instrument Shop and served as the standard to verify the calibration of the second L&N precision shunt. The second shunt will be used to periodically calibrate the commercial shunts located in the control frame.

* Each heater element is a pipe that is resistance-heated by current flowing through it.

○ - Shunt

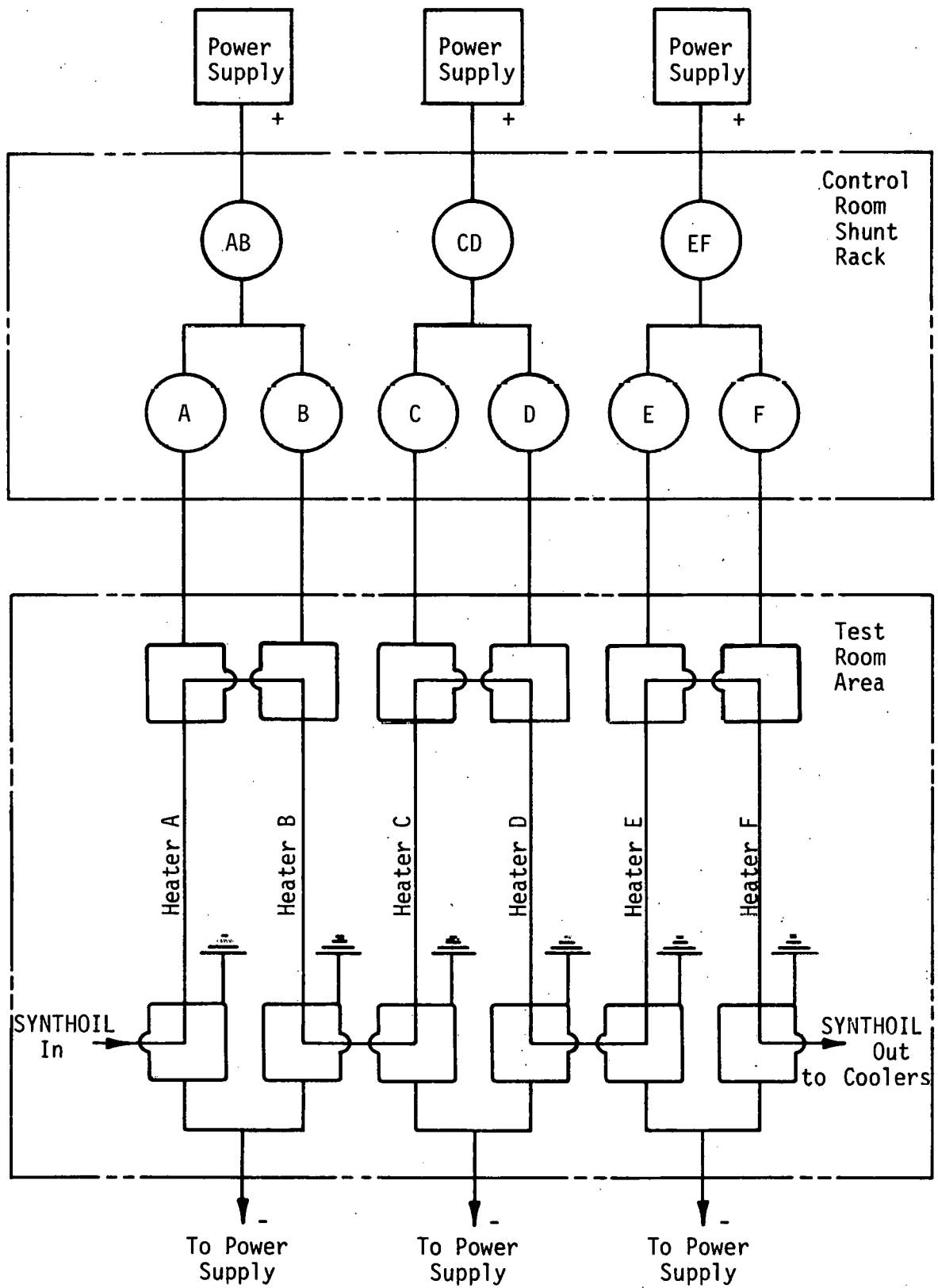


Fig. 6. Heating Element Electrical Schematic

Table 3. Shunt Values

Shunt Designation	Current, A	Nominal Resistance, m Ω
EF, E, F	200	1/4
CE, C, D	300	1/6
A, B	500	1/10
AB	750	1/15

Calibration tests were performed on four of the nine commercial shunts during verification testing of the calibration of the precision shunts to be used for periodic recalibration. The results indicated (1) variation in the calibration of the precision shunts was less than 0.05% and (2) the effect of temperature (i.e., when forced-air cooling was interrupted) on two of the commercial shunts was within acceptable limits. More detailed results are contained in Tables 4, 5, and 6.

Table 4. Calibration of Shunts E and EF.^a Test I

Current, A	Shunt Voltage, mV	Condition of F Shunt	$\overline{\Delta R}$, ^b m Ω	\overline{X} , ^c mV
93	23.2	cooled	0.00205	0.191
93	23.2	not cooled	0.00206	0.192
133	33	cooled	0.00208	0.277
180	45	cooled	0.00208	0.376
180	45	not cooled	0.00205	0.370
180	45	cooled	0.00207	0.373
201	50	cooled	0.00211	0.426
201	50	not cooled	0.00207	0.4165
201	50	cooled	0.00209	0.4218

^a Shunts E and EF have 200-A capacity

^b $\overline{\Delta R} = \frac{\overline{X}}{\text{current}}$

^c \overline{X} = mean difference of the voltages between the shunts.

Table 5. Calibration of Shunts D and CD.^a Test II

Current, A	Shunt Voltage, mV	Condition of D Shunt	$\overline{\Delta R}$, ^b m Ω	\overline{X} , ^c mV
87	14.4	not cooled	-0.00064	-0.056
94.7	15.6	cooled	-0.00062	-0.059
140	23.2	cooled	-0.00059	-0.082
181	29.9	cooled	-0.00059	-0.106
181	30.0	not cooled	-0.00064	-0.116
234	38.7	not cooled	-0.00060	-0.140
298	49.3	not cooled	-0.00065	-0.193
298	49.3	cooled	-0.00058	-0.174

^a Shunts D and CD have 300-A capacity.

^b $\overline{\Delta R} = \frac{\overline{X}}{\text{current}}$

^c \overline{X} = mean difference of the voltages between the shunts.

Table 6. Comparison of Precision Shunts

Current, A	Average % Deviation
<u>Test 1</u>	
94	-0.045
133	-0.005
180	-0.037
202	-0.04
<u>Test 2</u>	
94.7	+0.028
140	-0.042
181	+0.013
234	+0.004
298	-0.015

The data acquisition system that will be used to collect data from the heat transfer experiment was used to calibrate and verify shunt operation. The data acquisition system uses an integrating digital voltmeter as the monitoring element and can process two to three channels/sec of data. During the shunt calibration procedure, the data channels processing the precision shunt readings were adjacent, as were the channels processing the commercial shunt readings. The use of adjacent channels was expected to minimize the effect of short-term variations in the power supply output current. The detailed effects of the short-term current changes and the use of consecutive sampling on the variations noted in Tables 4 and 5 are not known.

Test I (Table 4) was for the calibration of shunts EF and F; Test II (Table 5) was for the calibration of shunts CD and D. In order to assess the effect of temperature changes on shunt calibration, the cooling air (electric fan) to a shunt was blocked; the voltage difference between the EF shunt and F shunt, X, was monitored to compare the differences in the presence and absence of cooling air flow. Table 4 is a summary of the calculated resistance differences, ΔR , using the mean difference of the voltages across the shunts.

It can be seen from Table 4 that the voltage differences between the cooled shunts and the uncooled shunts are very small. Since the resistance values of the EF and F shunts are about $1/4 \text{ m}\Omega$ (Table 3), the maximum changes (at 201 A) were less than 0.025% due to differences in temperature for the forced-cooling condition and the natural-convection condition. The change in shunt operating temperature between the forced-cooling condition and natural cooling condition was estimated at about 40°F .

Table 5 contains the information on comparison of the CD and D shunts during forced- and natural-convection cooling on the D shunt. Since the resistances of the D and CD shunts are $\sim 1/6 \text{ m}\Omega$ and since the effects of cooling are $<0.05\%$, the effect of temperature on the 300-A shunts can be neglected.

Table 7 contains the shunt calibration resistances and experimental standard deviation values.

Table 7. Shunt Calibration Values

Shunt	Resistance, $\text{m}\Omega$	Std Deviation
CD	0.16449	0.00015
D	0.16510	0.00016
EF	0.24816	0.00025
F	0.25024	0.00023

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2. J. Fischer *et al.*, "Coal Liquefaction Support Studies, Quarterly Report, October 1976-December 1976," Argonne National Laboratory, ANL-77-6.

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