STUDY OF EBULLATED BED FLUID DYNAMICS FOR H-COAL. QUARTERLY PROGRESS REPORT NO. 3, MARCH 1, 1978-MAY 31, 1978

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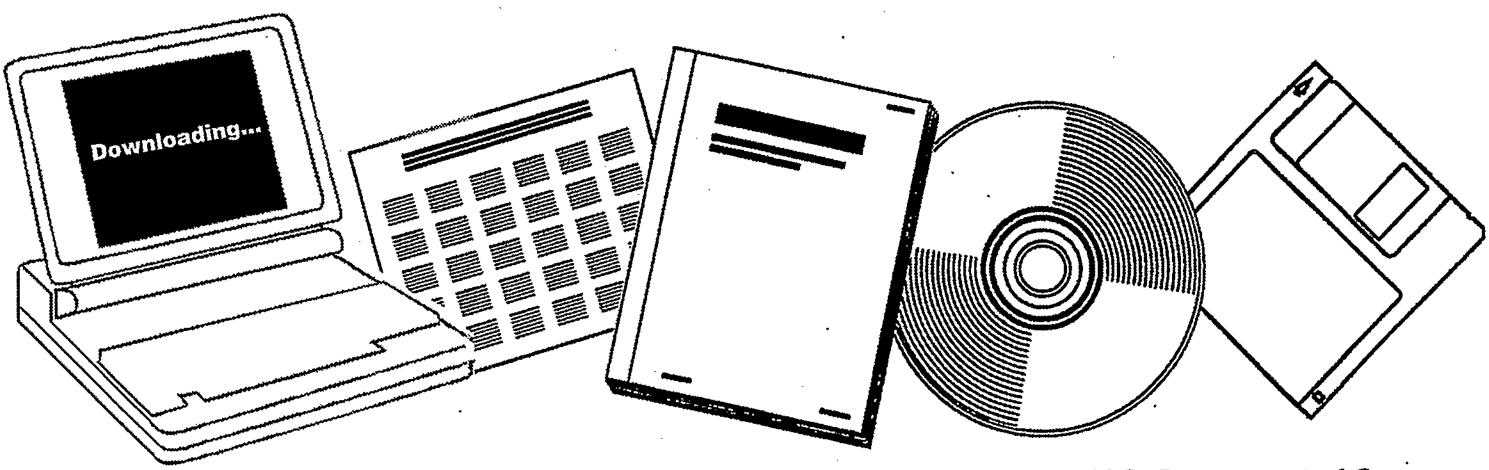

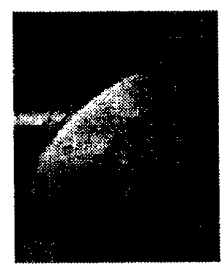

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STUDY OF EBULLATED BED FLUID DYNAMICS FOR H-COAL

QUARTERLY PROGRESS REPORT NO. 3
MARCH 1, 1978-MAY 31, 1978

I. A. VASALOS, E. M. BILD, T. D. EVANS,
D. F. TATTERSON, A. P. VANDER KLAY

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Research and Development Department

Amoco Oil Company
P. O. Box 400
Naperville, Illinois
60540

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FOREWORD

The H-Coal process, developed by Hydrocarbon Research, Incorporated (HRI), involves the direct catalytic hydroliquefaction of coal to low-sulfur boiler fuel or synthetic crude oil. The 200-600 ton-per-day H-Coal pilot plant is being constructed next to the Ashland Oil, Incorporated refinery at Catlettsburg, Kentucky under ERDA contract to Ashland Synthetic Fuels, Incorporated. The H-Coal ebullated bed reactor contains at least four discrete components: gas, liquid, catalyst, and unconverted coal and ash. Because of the complexity created by these four components, it is desirable to understand the fluid dynamics of the system. The objective of this program is to establish the dependence of the ebullated bed fluid dynamics on process parameters. This will permit improved control of the ebullated bed reactor.

The work to be performed is divided into three parts: review of prior work, cold flow model construction and operations, and mathematical modelling. The objective of this quarterly progress report is to outline progress in the first two parts during the third quarter of the project.

OBJECTIVE AND SCOPE OF WORK

The overall objective of this project is to improve control of the H-Coal reactor through a better understanding of the hydrodynamics of ebullated beds. The project is divided into three main tasks:

1) Review of prior work in three-phase fluidization.
2) Construction of a cold flow unit and collection of data.
3) Development of a mathematical model to describe the behavior of gas-liquid fluidized beds. The model will be based on information available in the literature and on data generated in the cold flow unit.

Progress made on Tasks 1 and 2 during the third quarter of the project is presented in this report.

SUMMARY OF PROGRESS TO DATE

Review of Prior Work

The draft report of the literature was revised based on comments received from H-Coal participants. The final report of the literature review issued on May 24, 1978.

Unit Construction and Data Collection

The design and construction of the fluid dynamics unit was completed. Unit debugging is almost complete.

Initial experiments in the unit were started using water and nitrogen.

Characterization of the fluids and solids which will be used in this experimental program continued this quarter. The surface tension of the liquids to be used in the cold flow unit were measured as a function of temperature. The particle size distribution of the coal-char particles to be used in making slurries in the project was also obtained. An attempt was also made to measure the viscosities of toluene and mineral oil slurries with a Brookfield Synchro-Lectric viscometer with a UL adapter. The viscosities were found to decline with time in the viscometer, indicating the coal-char particles were settling in the viscometer.

A liquid sampling system was designed to obtain liquid samples from HRI's PDU. HRI and Battelle were visited to obtain their comments and suggestions on the design. Parts for the system were ordered, and the system has been assembled.

The initial set of experiments to be carried out in the cold flow unit after debugging was planned.

Costs

By the end of the third quarter, about $360,000 was committed to the project. Future projections indicate that no cost overruns are anticipated at this time.

REVIEW OF PRIOR WORK

A draft report of the literature review was sent to H-Coal participants on March 16 for comments. The report was revised based on comments received and was issued May 24 to H-Coal participants.

CONSTRUCTION OF COLD FLOW UNIT AND DATA COLLECTION

Emphasis was placed on completing construction and debugging of the fluid dynamics unit. Work in all areas except shop construction was completed this quarter. Details on the construction and instrumentation of the pilot plant are given below.

Unit Construction

Following completion of the glass reactor assembly and installation of the feed tanks last quarter, piping was started on the unit this quarter. During March, gas piping was completed. This involved the installation of the gas compressor, gas-liquid separator, gas capacity tank, demister, knock-out drum, and the differential pressure taps and cells. Piping of liquid lines was also started. Screwed pipe fittings were used on the liquid lines. In conjunction with the liquid piping, the feed and recycle pumps were installed. Flow meters were not available and were piped into the unit as they were received.

Simultaneously with their installation, the analog outputs from the flow meters, pressure transmitters, and DP cells were wired into the panel board and recorders.

Installation of the gamma-ray elevator system was started in April. The gamma-ray source and scintillation detector are mounted on a steel structure, and the entire system is moved up and down the glass column using the elevator. The elevator is multi-speed and can be operated manually or via the computer system.

Piping on the unit was completed during April except for a few items which were delayed in delivery. Instrument wiring to the panel board continued during April and May. A plastic shield and guard railing were also installed around the reactor during these months.

· Leak testing of the unit started in April and continued into May. Gas lines were relatively leak-free; however, numerous leaks were found at the screwed fitting used in the liquid lines. These had to be redone, and in several instances the joints were welded to give leak-free connections. The glass reactor was tested with water and was found to be leak-free; however, three-way solenoid valves on the reactor bypass were not operating properly, so they were removed and realigned to prevent leakage through the valve.

The top glass section in the reactor shattered during the leak testing. The glass reactor is supported only from the bottom spool piece with lateral guides along its length. Since installation, the reactor had drifted far enough from its original position to be restricted by the guides, putting considerable stress on the glass and causing it to break. Following this incident, one lateral guide was removed from the reactor and some flexible connections were added to make sure the column could move freely without being put under stress. The position of the glass column relative to any supports will be carefully monitored to avoid putting any stress on the reactor.

The sample probe was tested on the unit, and modifications for the best operation were finalized. The probe will be marked to give its position in the reactor and to ease insertion and removal of the probe. The construction of the modified probe is the only shop construction remaining.

Instrument Installation

Instruments were installed on the fluid dynamics unit as they were delivered because, in general, these were the longest delivery items. An integral orifice meter is used to measure gas flow rate. The meter was calibrated on the unit using the off-gas dry test meter, and the calibration curve is shown in Figure 1. Initially, difficulty was encountered in obtaining steady readings from the integral orifice meter. It was repiped with straight pipe sections before and after the instrument, and this modification helped damp out the fluctuations.

The liquid slurry feed flow is measured using a Nusonics flow meter. The electronics of this meter are calibrated so the output is read directly in gallons per minute, so no calibration curve is necessary. We checked the electronics calibration by circulating water through the meter back to the feed tank on a scale. The meter reading was found to be accurate.

The liquid slurry recycle flow is monitored with a MicroMotion flow meter. This meter has both digital and analog outputs which read directly in pounds/minute, so again no calibration curve is necessary. The recycle lines were piped before the meter was delivered. When the meter was installed, it was discovered the analog output is sensitive to the flow direction through the meter. An engineer from MicroMotion modified the flow meter in situ so the unit piping would not have to be modified. The electronic calibration of this meter was checked, and both the digital and analog outputs were found to be accurate.

The pressure off the reactor top is measured with a Honeywell pressure transmitter. This transmitter was checked before and after installation on the unit and is calibrated for 0-15 psig.

The liquid level in the gas-liquid separator is measured using a Honeywell DP cell. Gas is purged through a dipleg in the tank, and the DP cell output gives the level of water in the tank in inches. For less dense liquids, a factor will be used to determine the liquid level.

The analog outputs from the three flow meters, the pressure transmitter, and the separator level LP cell go to the Honeywell DC 2000 control system. The transmitter outputs are displayed on recorders. Using this unit, all of these functions can be controlled at the desired set point.

The liquid level in the feed tank and the slurry preparation tank is monitored with the same DP cell system used for the separator except the outputs go to Photohelic gauges; thus, the level cannot be controlled.

Bournes DP cells located about 27" apart measure the pressure drop all along the reactor. The pressure measurements are recorded on Texas Instruments and Gould recorders. The overall pressure drop across the reactor is measured with a Honeywell DP cell.

A Nusonic concentration analyzer was installed in the feed to reactor in order to monitor the coal slurry concentration. The output of this meter is recorded on a Honeywell recorder.

The reactor gamma-ray scan system and electronics are located on the unit. A Harshaw scintillation detection system is used. The scan output is monitored using a Texas Instruments recorder on the panel board. Ten millicuries of Co-60 from K-Ray is used as the gamma-ray source. Using the elevator system, the reactor can be scanned continuously from the bottom spool piece to the recycle cup.

An Orteo scintillation detection system and a Cs-137 source are used to scan the liquid recycle line from the recycle cup at the bottom of the reactor. A vertical length of pipe is scanned. The system is used to monitor the quantity of entrained gas in the recycle liquid. The output is recorded with a Leeds & Northrup recorder.

Both of the gamma-ray systems were calibrated to give optimum sensitivity to density changes. A scan of the reactor filled only with water is shown in Figure 2b. The spikes show the location of the spool pieces. The reactor is scanned in both directions. Figure 2a shows a scan with both water and gas flowing through the reactor. The addition of gas resulted in greater transmission of gamma-rays, and thus a higher count rate, through the reactor.

A Rosemount pressure transmitter is connected in the discharge line of the slurry recycle pump. The discharge pressure is monitored with a Honeywell recorder. The output is related to the amount of entrained gas in the slurry recycle.

The compressor discharge pressure is detected with a Foxboro pressure transmitter and recorded on a Leeds & Northrup Azar recorder.

A Beckman oxygen analyzer was placed in the recycle gas line to assure that oxygen is not leaking into the compressor when combustible liquids are being used in the reactor.

The pressure transmitters and oxygen analyzer were checked for operability and accuracy after installation on the unit.

Data Collection

Physical Properties.--During the quarter, preliminary data collection started. The surface tension of liquids to be used in the cold flow unit were measured as a function of temperature. These results are reported in Table I. A Fisher Scientific surface tensiometer (Model 20) was used in these measurements. The results indicate that surface tension varies linearly with temperature over the range of interest.

The cumulative size distribution of the coal-char particles which will be used to make slurries was measured by IIT Research Institute using an optical microscope interfaced with a Quantimet 720 computerized image analyzer. The distribution is reported in Table II. The geometric mean particle diameter is 3.4 microns.

HDS-2A catalyst to be used in the cold flow studies has been obtained. The lengths of catalyst chosen for study are 1/8", 3/16", 1/4", and 3/8"; the diameter of the catalyst is 1/16". Cyanamid has guaranteed that the variation of length will not exceed ± 20% for each of the above lengths. The length distribution for the 1/8", 3/16", 1/ 1/4", and 3/8" long catalyst is listed in Table III.

The viscosity of toluene and mineral oil slurries containing 7 and 14 vol% coal-char fines was measured with a Brookfield Synchro-Lectric viscometer with a UL adapter. The diameters of the spindle and cup for this adapter are approximately 2.5 and 2.7 cm, respectively. The viscosities of these slurries were found to decline with time in the viscometer. This result indicates that the coal-char particles are settling out in the viscometer.

A capillary tube viscometer having an ID of 2.1 mm and a length of 100 cm is being constructed to measure the viscosity of kerosene and mineral oil slurries. This viscometer can be used either in the lab or on the cold flow unit to measure slurry viscosity.

Unit Data.--As an aid to unit check-out and debugging, several runs were made with water and nitrogen at several flow rates. The combination of flow rates used is given in Table IV. Data were collected from the DP cells along the reactor, and gamma-ray scans of the reactor were obtained for each test. The data are shown in Table V. The DP measurements were used to calculate liquid void fraction for the eight sections of the reactor and the reactor average. The gamma-ray scan data were used to calculate a reactor average of the liquid void fraction. These results are shown in Table IV. In general, the average liquid hold-up in the reactor calculated using DP measurements and the gamma-ray data decreased with increasing gas flow rate, as would be expected. However, considerable fluctuations in the DP readings led to difficulty in estimating the average DP at each location in the reactor. So, although changes in overall liquid void fractions appear reasonable, there is considerable question on the accuracy and reliability of individual DP measurements. Solutions to the unstable readings were being sought. Gas-purged taps are currently being used. Liquid-filled taps should dampen vibrations considerably. Also, needle valves could be put in the gas impulse lines to dampen out fluctuations. Both of these solutions will be checked during the next month.

Gamma-ray scans of the reactor worked well, and the data will be even more reliable when the elevator is operated using the computer. Computer operation of the elevator should commence early next month.

Measurement of Physical Properties of H-Coal Liquids

A sampling system has been designed and constructed to obtain liquid samples from HRI's PDU. HRI and Battelle Labs were visited to obtain their comments and suggestions on the design. The final design is shown in Figure 3. It is designed to operate at temperatures up to 900°F and 3000 psig. A detailed description of the operating procedure is given in March's Monthly Progress Report.

During the HRI meeting, it was also decided that the sample point would be between the external separator and the first-stage pressure letdown valve before the atmospheric flash. HRI will install a sampling point which will include two block valves.

During the Battelle meeting it was also decided that hydrogen will be used to transfer the liquid from the sample vessel into the viscometer. Hydrogen will also be used to pressurize the viscometer during measurements. Nitrogen will be substituted for hydrogen in a few measurements at low temperature to determine the effect of gas type on viscosity.

Battelle also pointed out that they expect coking (polymerization) problems to develop with the H-Coal samples at high temperatures (300-400°C). This expectation is based on their previous experience in measuring the viscosity of Synthoil samples. In these measurements it was found that at high temperatures, viscosity changed with time, thus indicating that the sample is physically changing in some manner.

Battelle has submitted a proposed contract to Amoco for measurement of the viscosity of the H-Coal liquid samples. The viscosity of four samples will be measured over the temperature range of 370-480°C at pressures ranging from 2000 to 3000 psi hydrogen. The proposed starting date for the contract is July 1, 1978. The cost is $17,390.

EXPERIMENTAL PLAN

The initial set of experiments to be performed in the unit after debugging was planned. The combination of variables--gas, liquid, volume per cent fines, and particle l/d ratio of the catalyst--to be used for each run is listed in Table VII. Gas and liquid flow rates in these experiments will be varied over the full range for which the unit was designed.

The results from these tests will indicate significant effects and give estimates of interaction effects for the above variables. Interaction between these variables can be determined by additional runs if the results of these experiments indicate that it is desirable or necessary to do so.

COSTS

By the end of the third quarter, approximately $360,000 was spent in the total completion of the literature search and the partial completion of the construction of the H-Coal fluid dynamics unit. As shown in Table VIII, the total cost of the unit is estimated at this time at about $361,500. A large part of this cost has already been incurred. However, due to material procurement delays, some of these costs will appear during the next quarter. Today, every indication is that project costs are on target. Future cost projections for the project life are shown in Figure 4.

FUTURE PLANS

During the next quarter the following are planned:

1) Complete wiring of instruments to the computer.

2) Debug computer hardware and software.

3) Complete initial experiments with water, nitrogen, and catalyst.

4) Start experimental program to determine significant effects.

5) Analyze data from the above experiments and use to test correlations and models in the literature.

6) Ship PDU liquid sampling system to HRI and obtain liquid samples during an appropriate PDU run.

TABLE I

SURFACE TENSION OF FLUIDS
TO BE USED IN H-COAL UNIT, DYNES/CM

	50°F	74°F	150°F
Water	77.6	75.5	68.3
Toluene	31.7	30.8	24.5
Kerosene	30.0	28.6	24.6
Mineral Oil	34.0	32.6	29.1

DFT/ml
4/4/78

TABLE II

CUMULATIVE SIZE DISTRIBUTION OF COAL CHAR

CUMULATIVE NUMBER AND NUMBER %
GREATER THAN STATED SIZE

Size, μm	Cumulative Number	Cumulative Number %
0	2916	100
1.1	2635	90.4
2.7	1921	65.9
3.8	1396	47.9
5.4	922	31.6
8.1	529	18.1
13.5	212	7.3
18.7	115	3.9
29.7	64	2.2
51.3	24	0.8
72.8	13	0.4
94.4	8	0.3

TABLE III

LENGTH DISTRIBUTIONS OF HDS-2A CATALYST
TO BE USED IN COLD FLOW EXPERIMENTS

Nominal Length = 1/8"

Length (Inches)	Number
Below 0.095	9
0.095-0.100	6
0.101-0.105	6
0.106-0.10	8
0.111-0.115	10
0.116-0.120	11
0.121-1.125	14
0.126-0.130	4
0.131-0.135	5
0.136-0.140	2
0.141-0.145	2
0.146-0.150	1
Above 0.150	1

Nominal Length = 3/16"

Length (Inches)	Number
Below 0.150	1
0.150-0.1575	0
0.1576-0.1650	2
0.1651-0.1725	6
0.1726-0.1800	9
0.1801-0.1875	5
0.1876-0.1950	4
0.1951-0.2025	7
0.2026-0.2100	9
0.2101-0.2175	4
0.2176-0.2250	
0.2351-0.2325	1
0.2326-0.2400	3
0.2401-0.2475	4
Above 0.2475	5

Nominal Length = 1/4"

Length (Inches)	Number
Below 0.200	2
0.200-0.210	2
0.211-0.220	7
0.221-0.230	3
0.231-0.240	2
0.241-0.250	7
0.251-0.260	5
0.261-0.270	8
0.271-0.280	7
0.281-0.290	3
0.291-0.300	3
0.301-0.310	3
Above 0.310	8

Nominal Length = 3/8"

Length (Inches)	Number
Below 0.300	3
0.300-0.315	6
0.316-0.330	5
0.331-0.345	10
0.346-0.360	8
0.361-0.375	10
0.376-0.390	7
0.391-0.405	4
0.406-0.420	9
0.421-0.435	5
0.436-0.450	4
0.451-0.465	3
Above 0.465	6

DFT/ml
6/13/78

TABLE IV

EXPERIMENTAL TEST CONDITIONS

Test No.	Liquid Flow Rate, GPM/Ft^2	Gas Flow Rate, Ft/Sec
1a	22.4	0.05
1b	22.4	0.10
1c	22.4	0.15
2a	44.8	0.05
2b	44.8	0.10
2c	44.8	0.15
2d	44.8	0.20
3a	67.2	0.05
3b	67.2	0.10
3c	67.2	0.15

EMB/ml
6/13/78

TABLE V

EXPERIMENTAL DATA FROM DP CELLS AND GAMMA-RAY SCANS

	1a	1b	1c	2a	2b	2c	2d	3a	3b	3c
DP1 (Bottom Spool Piece-27")(In. of H_2O)	23	22.8	20	22	21	19	19	22	20	17
DP2 (27-54")(In. of H_2O)	22	22	22	22	21	25	30	22	22	27
DP3 (54-81")(In. of H_2O)	22	20	12	22	22	11	11	23	13	11
DP4 (81-108")(In. of H_2O)	22	24	22	19	22	22	23	22	26	26
DP5 (108-135")(In. of H_2O)	24	24	20	24	22	20	18	23	20	18
DP6 (135-162")(In. of H_2O)	23	24	22	21	22	22	20	22	22	22
DP7 (162-189")(In. of H_2O)	22	22	20	22	22	21	20	22	22	21
DP8 (189"-Top of Recycle Cup-3.5 Ft) (In. of H_2O)	33	38	36	30	36	38	40	26	33	42
Gamma-Ray Scan Reactor Average (Counts/Sec)	260	260	271	240	250	260	281	260	271	281

EMB/ml
6/13/78

TABLE VI

CALCULATED LIQUID VOID FRACTIONS FROM DP AND GAMMA-RAY SCAN DATA

	1a	1b	1c	2a	2b	2c	2d	3a	3b	3c
E_{l_1}*	0.85	0.85	0.76	0.83	0.78	0.71	0.71	0.83	0.78	0.65
E_{l_2}	0.84	0.84	0.84	0.82	0.82	0.93	1.0	0.82	0.82	1.0
E_{l_3}	0.82	0.75	0.45	0.82	0.64	0.41	0.41	0.85	0.48	0.41
E_{l_4}	0.82	0.89	0.82	0.71	0.82	0.82	0.86	0.82	0.97	0.97
E_{l_5}	0.89	0.89	0.74	0.90	0.82	0.75	0.67	0.85	0.75	0.67
E_{l_6}	0.86	0.89	0.82	0.78	0.82	0.82	0.75	0.82	0.82	0.82
E_{l_7}	0.84	0.82	0.77	0.84	0.84	0.78	0.73	0.82	0.82	0.78
E_{l_8}	0.76	0.89	0.86	0.71	0.82	0.89	0.95	0.62	0.78	0.99
E_l avg (Gamma Scans)	0.84	0.84	0.81	0.92	0.88	0.84	0.79	0.84	0.81	0.79

*Subscripts indicate which DP cell reading was used to calculate the void fraction.

EMB/ml
6/13/78

TABLE VII

INITIAL EXPERIMENTS

Run No.	Fines (Vol%)	l/d	Liquid	Gas
1*	0	2	Kerosene	Helium
2	15	2	Kerosene	Freon-12
3	0	6	Kerosene	Freon-12
4*	15	6	Kerosene	Helium
5*	0	2	Mineral Oil	Freon-12
6	15	2	Mineral Oil	Helium
7	0	6	Mineral Oil	Helium
8*	15	6	Mineral Oil	Freon-12

*Runs to be replicated.

DFT/ml
5/4/78

TABLE VIII

H-COAL FLUID DYNAMICS
DESIGN AND CONSTRUCTION COSTS

A) Materials	$137,000
B) Craft Labor	
1977 Charges	6,800
Electrical	14,000
Pipefitting	25,000
Sheet Metal	6,200
Special--ARC	13,000
	$ 65,000
C) Miscellaneous	
Professional and Support Staff	57,800
Professional--Mechanical	17,000
Professional--Systems	54,700
Professional--Construction Coordination	10,000
	$139,500
D) Computer Software Development	$ 20,000
TOTAL	$361,500

IAV/ml
6/13/78

Figure 1

CALIBRATION OF THE INTEGRAL ORIFICE METER

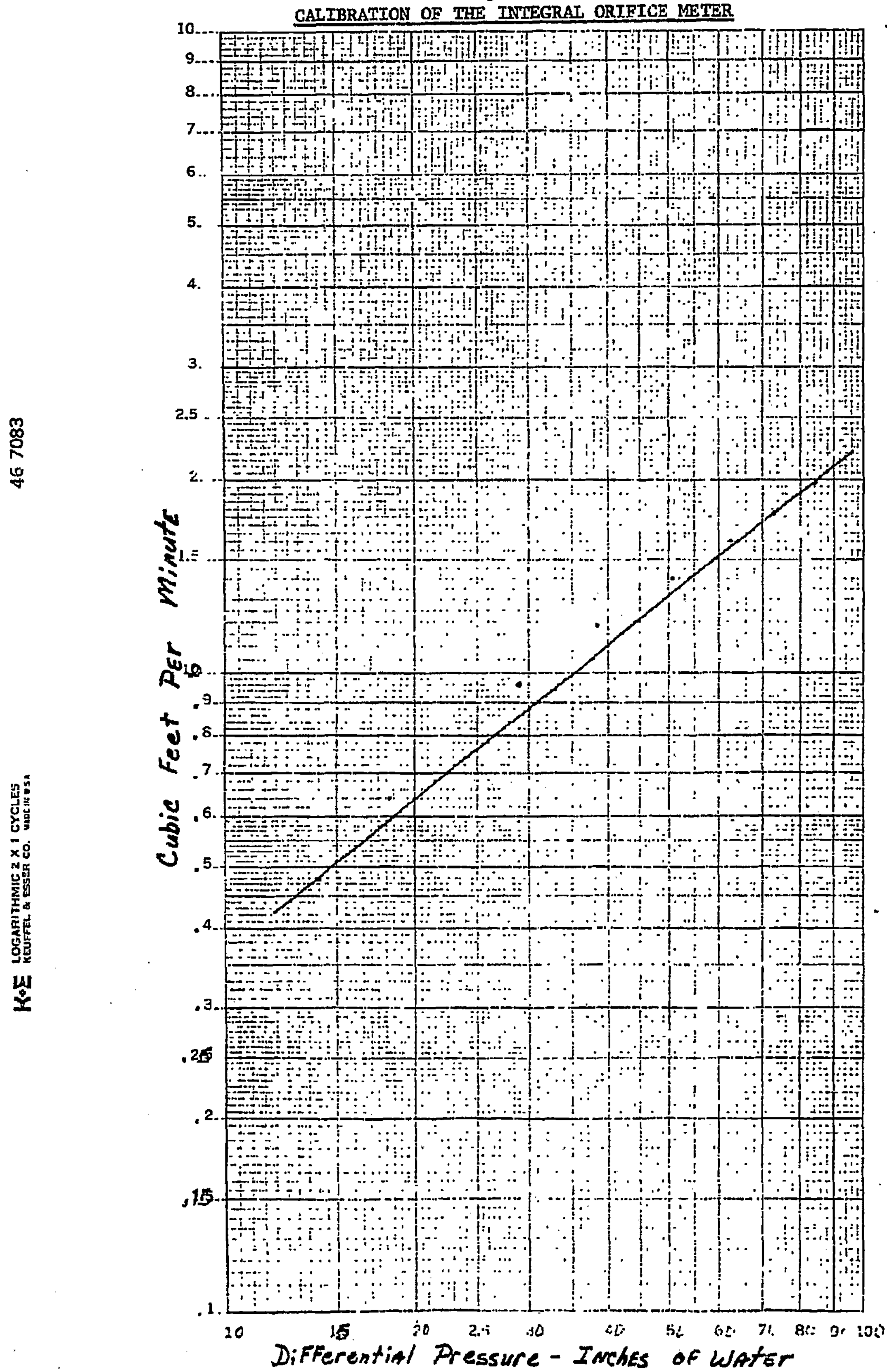
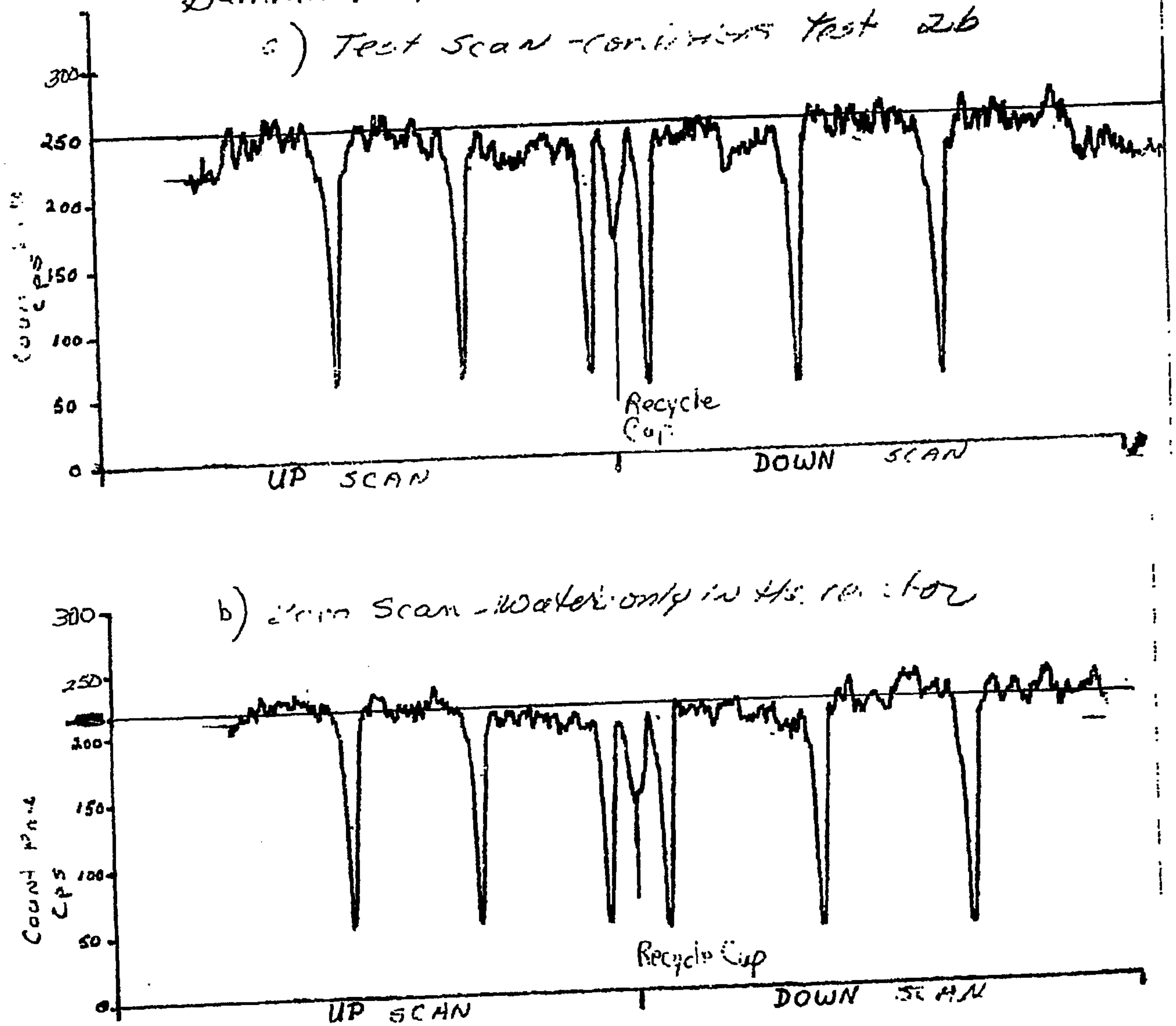


Figure 2
Gamma-Ray Scans of the Glass Reactor



PDU SAMPLING SYSTEM

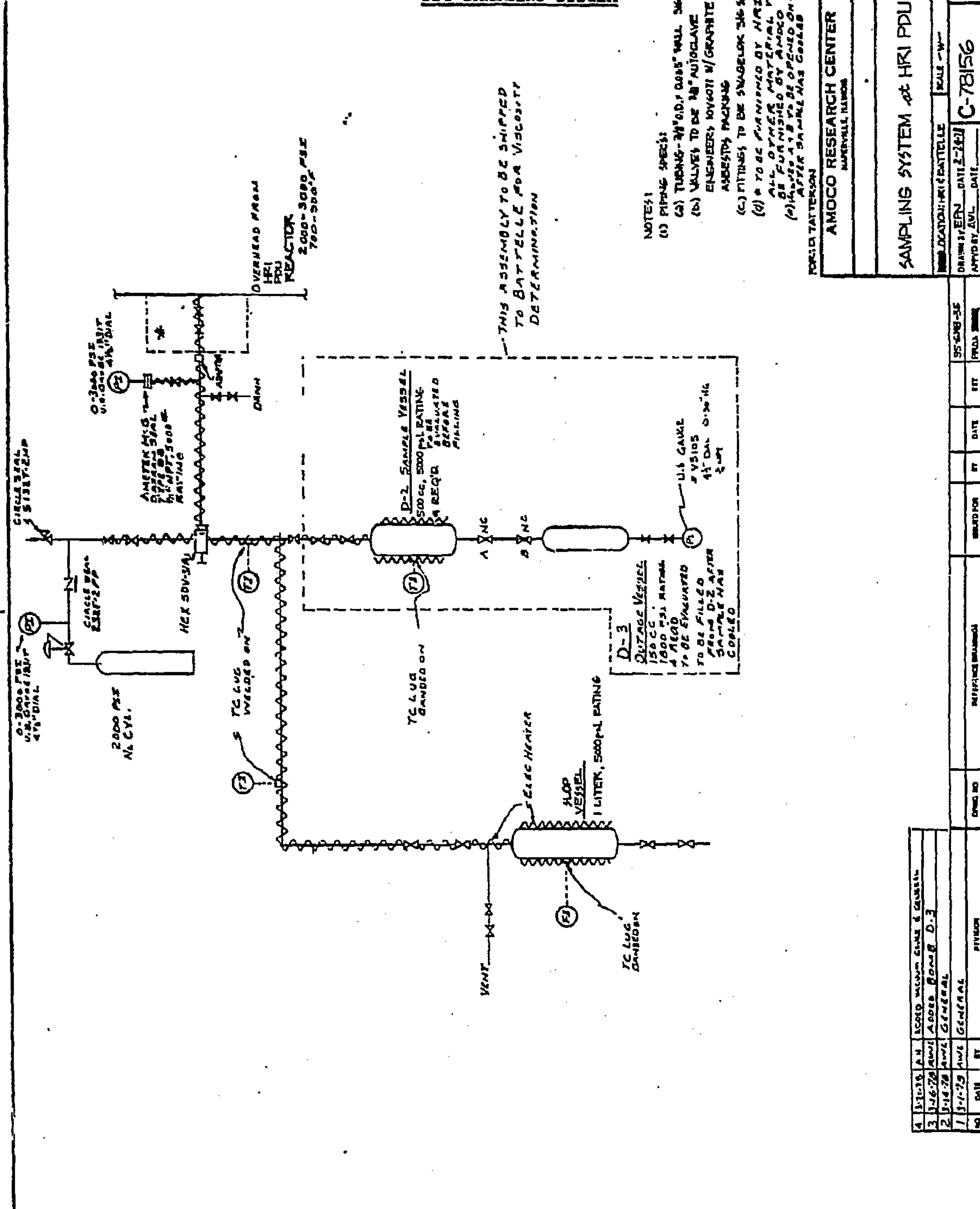
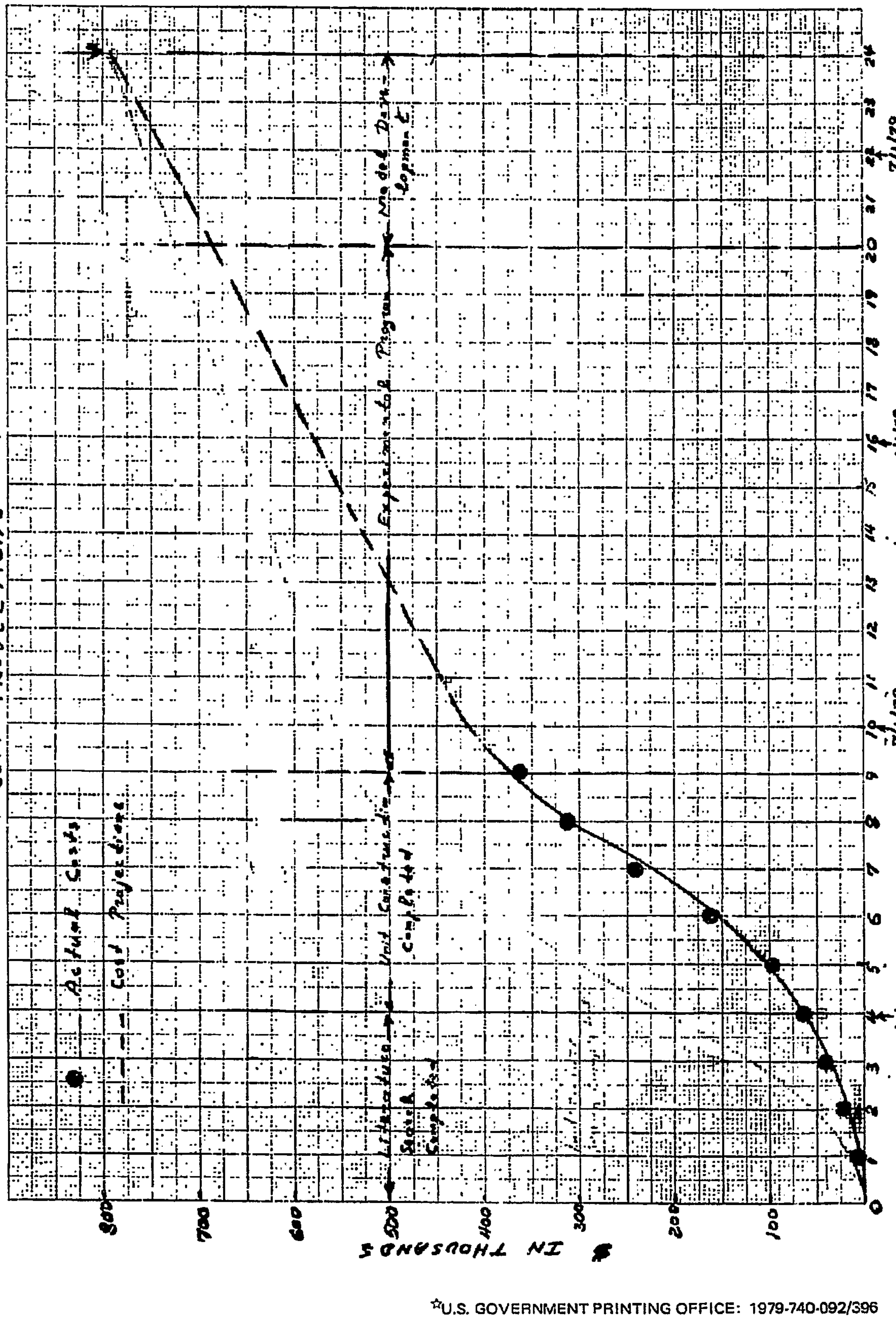


Figure 4 — COST PROJECTIONS



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