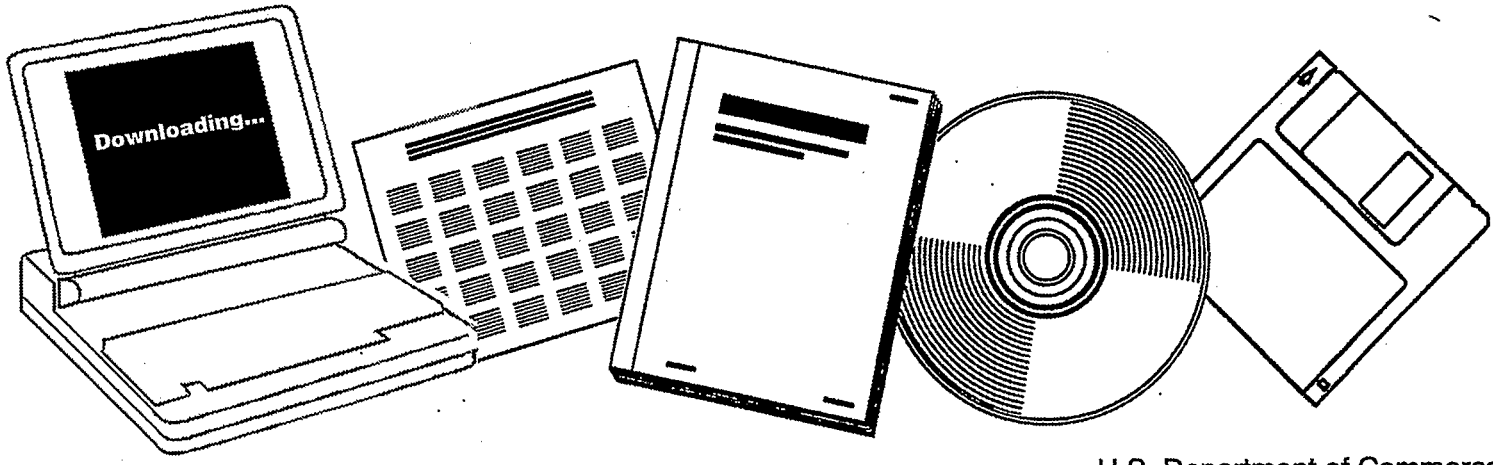
STUDY OF EBULLATED BED FLUID DYNAMICS FOR H-COAL. QUARTERLY PROGRESS REPORT NO. 1, JULY 1-SEPTEMBER 30, 1980

AMOCO OIL CO., NAPERVILLE, IL. RESEARCH AND DEVELOPMENT DEPT

DEC 1980



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STUDY OF EBULLATED BED FLUID DYNAMICS FOR H-COAL

QUARTERLY PROGRESS REPORT NO. 1
JULY 1-SEPTEMBER 30, 1980

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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 operated next to the Ashland Oil, Incorporated, refinery at Catlettsburg, Kentucky, under DOE 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. One objective of this program is to apply the results of prior cold flow model experiments (1) to the operating H-Coal PDU reactor in Trenton, New Jersey. Studies are also planned to examine the coalescence behavior of gas bubbles in three-phase ebullated beds.

The work to be performed is divided into four parts: fluid dynamics measurements on the PDU reactor, gas bubble coalescence studies at Northwestern University, cold flow and mixing tests at Amoco's Naperville Research Center, and model implementation. The objective of this quarterly progress report is to outline progress in the first three areas.

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SUMMARY

Cold flow experiments were completed with kerosene, nitrogen, and HDS-2A (3/16" length) catalyst. Per cent bed expansion, gas/liquid/catalyst holdups, and drift fluxes were determined for each test.

Fluid dynamics data were obtained at HRI during Run PDU-10 (Wyodak coal and Amocat-1A catalyst). Reactor liquid samples were taken for later viscosity determination.

A 6" diameter test stand for bubble coalescence experiments was constructed and delivered to Northwestern University. A search was initiated to select suitable model fluids.

INTRODUCTION

The fluid dynamics of the H-Coal reactor have been previously studied in a cold flow unit. Reference 1 provides details of the construction of the unit and results of tests with a variety of gases, liquids, and catalyst sizes. A semi-theoretical model was developed to predict the volume fractions occupied by the gas, liquid, and catalyst phases. The aims of this new contract are fourfold:

1) The model developed using cold flow unit test results will be extended to apply to the operating H-Coal PDU reactor.

2) Because gas bubble dynamics are crucial in determining the nature of the flow, studies of bubble flow will be performed at Northwestern University using optically clear beds.

3) Liquid mixing tests will determine the residence time distribution of liquid in the reactor. Under the previous contract, it was determined that the coal char fines (simulating the unreacted coal and ash) were uniformly distributed throughout the bed. Hence, the measurement of liquid data is essential for modeling the residence time and kinetic parameters associated with the unreacted coal.

4) The model will be implemented into a readily usable format.

DATA COLLECTION

HRI PDU Fluid Dynamics Study

A series of fluid dynamics tests was performed on the HRI PDU reactor to measure the response of component volume fractions to changes in the operating conditions. Data from these tests will also be used to validate the Bhatia-Epstein model developed under a previous contract. These data were obtained by measuring the absorption of gamma-rays passing through

the reactor along its entire length. Auxiliary experiments were performed to measure the absorption of the empty PDU reactor before the run and of the settled catalyst bed at the end of the run. Sixteen liquid product samples were taken for viscosity measurement.

Gamma-Ray Scan.--A gamma-ray scintillation detector was mounted on the existing PDU reactor traverse scanner. The 1" x 1" detector crystal was positioned opposite the center of the beam from a 500 mc Cs-137 source. This installation was designed to retain the existing HRI bed density monitor equipment.

Side and top views of the assembly appear in Figures 1 and 2, respectively. A schematic description of the electronics train which was used appears in Figure 3. The criteria for setting the gain of the amplifier and discriminator to bracket the Cs-137 photopeak are presented in Figure 4. Because the apparent peak energy may shift with detector temperature (Figure 5), returning is necessary before each test.

The first scan to be performed is a zero scan on an empty reactor. The no-flow gamma-ray absorption and that under operating conditions are used to calculate the average density at each point. Knowledge of the catalyst bed expansion and liquid density will then allow calculation of all component phase volume fractions.

On July 11, 1980, the empty reactor (zero) gamma-ray scan was taken. After tuning the detector electronics, absorption measurements were obtained at 2" intervals, with some 1" data near the bottom of the column. Gamma-ray scans on the operating PDU were taken at fourteen test points during the run. Table I presents a summary of the test dates, HRI period numbers, and approximate operating conditions.

Three scans were also taken during shutdown (9/24 AM, 9/24 PM, and 9/25 AM) to measure the absorption of the settled catalyst bed.

Viscosity Sampling Procedure.--HRI modified the previous method of collecting reactor liquid viscosity samples. Details of the new procedure appear in Appendix A. Sixteen viscosity samples were taken as detailed in Table II. During the test period, four samples were also withdrawn for Oak Ridge National Laboratory (ORNL) viscosity and density measurement.

Northwestern University Bubble Dynamics Study

The cold flow test stand was fabricated at Amoco and delivered to Northwestern. Figure 6 presents a schematic P+I diagram of the unit. It is designed to duplicate the main features of the cold flow pilot plant at Naperville, but yet be sufficiently simple to allow experimenters to concentrate on the laser holographic experiments rather than on equipment maintenance.

Bench-scale tests with toluene/safflower oil mixtures identified some problems with this choice of fluids. Dissolution of the UVA-7 acrylic plastic which simulated the catalyst particles was severe at the higher concentrations of toluene required to match the optical densities. Therefore, a new approach is under investigation. The catalyst pellets will be simulated by cylindrical pieces of glass rod (index of refraction = 1.474), and the liquid will be a mixture of dipropylene glycol monomethyl ether (Dowanol DPM) and diphenyl ether (a component of DowTherm heat transfer fluids). In a 66%/34% mixture, the viscosity is projected to be about 3.5 cp. A progress report from Northwestern University is given in Appendix B.

A safety review is under way now to determine any precautions necessary for use of these fluids.

Cold Flow Unit Data Collection

Using HDS-2A catalyst, an initial series of tests (Run 218) was performed to measure unit performance and provide a base line for future tests. Kerosene and nitrogen were the fluidizing liquid and gas, respectively. No coal char fines were present.

RESULTS

HRI PDU Fluid Dynamics Tests

The results of the zero gamma-ray scan taken on the empty reactor are displayed in Figure 7. At each location, the count rate displayed is the average of at least two separate measurements. Standard deviations were on the order of $\pm$ 1%. At elevations of 4' and 6', sharp drops in the average zero count rate may be seen. These were caused by external pipes and flanges connected to the reactor. Data taken here will be most sensitive to possible errors in traverse position during operating test scans. Therefore, results from these two locations may possibly not be used.

Count rates of the gamma-ray scan taken during PDU operation appear in Figures 8-15. These data will be used to calculate the volumes occupied by the gas, liquid, and catalyst at each point of the reactor height.

Cold Flow Model Fluid Dynamics

Summaries of catalyst bed expansions, catalyst bed (dense phase) component holdups, and holdups in the dilute phase above the bed are given in Tables III-V, respectively.

Liquid/Solid Fluidization.--Data for the liquid/solid tests (Runs 218-1 through 218-6) were analyzed using the Richardson-Zaki correlation:

$$\varepsilon_l^n = U_l/U_t$$

Liquid holdups may be determined by gamma-ray absorption ($\varepsilon_{l\gamma}$) or by calculations using the observed catalyst bed expansion ($1 - \varepsilon_c$). Both variables are plotted versus U_l in Figure 16. The Richardson-Zaki parameters are presented in Table VI. The extrapolated terminal velocity is similar with both data treatments. The slight discrepancy in liquid holdups as measured by these two techniques causes a significant shift in the exponent n. both parameters agree well with those for two other experiments (Table VII).

Gas/Liquid/Solid Fluidization.--The expansion of the catalyst bed at various liquid and gas flows is one parameter for describing ebullated bed operation. As seen in Figure 17, the catalyst bed continues to expand with gas flow. This behavior was noted in several other experimental runs (previously reported in Reference 1) where kerosene without slurried fines was used as the fluidizing liquid.

Three-phase data may be correlated using the drift flux approach of Darton and Harrison. The drift flux V_{CD} is defined as:

$$V_{CD} = U_g(1 - \varepsilon_g) - U_l\varepsilon_g(1 - \varepsilon_g)/(\varepsilon_l + \varepsilon_f)$$

Data points from the current experiments may be contrasted with those from a previous test, Run 212, reported in Reference 1. In Figure 18, this comparison also includes the previously obtained relation between V_{CD} and ε_g defining the ideal bubbly regime. A slightly larger drift flux slope is seen for the Run 218 experiments; however, no departures into the transition regime or churn turbulent behavior are apparent.

PLANS FOR NEXT PERIOD

Task 1: H-Coal PDU Reactor Fluid Dynamics

1) Begin analysis of the gamma-ray scan data. Compute the average mass absorption coefficients for liquid and catalyst.

2) Begin viscosity measurements at Battelle Institute.

3) Begin analytical characterization of slurry mix tank samples.

Task 2: Gas Bubble Dynamics (Northwestern University)

1) Continue assembly and testing of transparent bed system, including optical components.

Task 3: Amoco Fluid Dynamics tests

No further tests with HDS-2A catalyst are planned at this time. At the end of the PDU-10 run, Amocat-1A samples may be obtained and tested. Large-scale production of Amocat-1A to support operations at Catlettsburg

is scheduled for early 1981. Selected tests, both with and without coal char fines, may be performed at that time using this catalyst.

Task 4: User Model

No work during the next period is planned in this area.

REFERENCES

1) Vasalos, I. A., et al., "H-Coal Fluid Dynamics: Final Report," DOE Contract DE-AC05-77ET-10149, published February, 1980.

NOMENCLATURE

U_g	Superficial gas velocity, ft/sec
U_l	Superficial liquid velocity, ft/sec
U_t	Catalyst particle terminal velocity, ft/sec
V_{CD}	Drift flux, mm/sec
ϵ_c	Catalyst volume fraction
ϵ_f	Fines volume fraction
ϵ_g	Gas volume fraction
ϵ_l	Liquid volume fraction

Additional Subscripts on Volume Fractions

B	Bed
GB	In bed, determined by gamma ray
DPB	In bed, determined by pressure drop
G	Above bed, determined by gamma ray
DP	Above bed, determined by pressure drop

TABLE I

PDU TEST SCHEDULE SUMMARY

Date (1980)	Period	Slurry Feed Rate, Lb/Hr*	Slurry Recycle Rate, GPM/Ft^2	Makeup H_2 SCFH	Recycle Gas SCFH
7/30	04A	822	47.1	3280	4178
8/05	10A	809	16.5	3485	4171
9/04	27A	606	35.9	2949	4330
9/11	34A	621	18.5	2935	4389
9/19A	41B	699	41.5	2850	4550
9/19P	42A	644	29.5	2900	4470
9/20A	42B	636	19.1	2860	4500
9/20P	43A	637	18.8	3610	6010
9/21A	43B	628	19.1	2830	2200
9/21P	44A	639	30.5	3940	6360
9/22A	44B	641	30.5	2790	2350
9/22P	45A	660	37.1	3690	6390
9/23A	45B	623	41.2	2850	2100
9/23P	46A	696	41.2	2820	4340

*Dry coal basis.

DNR/ml
12/31/80

TABLE II

PDU VISCOSITY SAMPLE SCHEDULE

Sample ID		Period Sample Was Taken	Date
Amoco-1		130-93-04A	7/30/80
-2		130-93-04A	7/30/80
-3		130-93-10A	8/05/80
-4		130-93-20A	8/28/80
-5	ORNL-1	130-93-27A	9/04/80
-6	ORNL-2	130-93-34A	9/11/80
-7		130-93-41B	9/19/80 AM
-8		139-93-42A	9/19/80 PM
-9	ORNL-3	130-93-42A	9/19/80 PM
-10		130-93-42B	9/20/80 AM
-11	ORNL-4	130-93-43A	9/20/80 PM
-12		130-93-43B	9/21/80 AM
-13		130-93-44A	9/21/80 PM
-14		130-93-44B	9/22/80 AM
-15		130-93-45A	9/22/80 PM
-16		130-93-45B	9/23/80 AM

DNR/ml
12/31/80

TABLE III

% BED EXPANSION FOR RUN 218

CATALYST : HDS-2A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 0.0 VOL %
TEMPERATURE : 76. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate Ft/Sec	Catalyst Bed Height (In.)	% Bed Expansion
218- 1	0.07	0.0	56.	12.
- 2	0.09	0.0	60.	20.
- 3	0.10	0.0	63.	26.
- 4	0.13	0.0	72.	44.
- 5	0.15	0.0	80.	60.
- 6	0.17	0.0	90.	80.
- 7	0.09	0.05	65.	30.
- 8	0.09	0.10	70.	40.
- 9	0.09	0.13	72.	44.
-10	0.09	0.16	72.	44.
-11	0.09	0.19	72.	44.
-12	0.10	0.06	67.	34.
-13	0.10	0.10	75.	50.
-14	0.10	0.14	80.	60.
-15	0.10	0.20	82.	64.
-16	0.15	0.05	84.	68.
-17	0.15	0.11	92.	84.
-18	0.15	0.16	102.	104.
-19	0.17	0.05	97.	94.
-20	0.15	0.22	109.	122.
-21	0.17	0.11	102.	108.
-22	0.17	0.17	111.	127.
-23	0.20	0.05	112.	129.
-24	0.20	0.12	116.	137.

TABLE IV

CALCULATED HOLDUPS, RUN 218: DENSE PHASE

CATALYST : HDS-2A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 0.0 VOL %
TEMPERATURE : 76. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ECB	ELGB	ELDPB	EGB	Vcd (Mm/Sec)
218- 1	0.069	0.0	0.44	0.52	0.64	0.0	0.0
- 2	0.086	0.0	0.41	0.55	0.64	0.0	0.0
- 3	0.101	0.0	0.39	0.58	0.65	0.0	0.0
- 4	0.126	0.0	0.34	0.62	0.69	0.0	0.0
- 5	0.149	0.0	0.31	0.68	0.72	0.0	0.0
- 6	0.173	0.0	0.27	0.71	0.74	0.0	0.0
- 7	0.087	0.048	0.38	0.57	0.51	0.05	11.5
- 8	0.087	0.099	0.35	0.51	0.46	0.14	20.0
- 9	0.087	0.132	0.34	0.49	0.43	0.17	25.3
-10	0.087	0.160	0.34	0.47	0.41	0.19	31.2
-11	0.087	0.193	0.34	0.45	0.40	0.21	36.4
-12	0.102	0.056	0.37	0.57	0.50	0.06	13.0
-13	0.102	0.100	0.33	0.54	0.48	0.13	19.9
-14	0.101	0.143	0.31	0.50	0.47	0.19	25.9
-15	0.101	0.200	0.30	0.47	0.47	0.23	35.6
-16	0.151	0.047	0.29	0.65	0.61	0.06	9.2
-17	0.150	0.108	0.27	0.61	0.58	0.13	20.3
-18	0.151	0.164	0.24	0.56	0.55	0.20	26.5
-19	0.172	0.048	0.25	0.69	0.66	0.06	9.6
-20	0.153	0.223	0.22	0.53	0.53	0.25	34.5
-21	0.172	0.113	0.24	0.68	0.62	0.09	25.1
-22	0.172	0.169	0.22	0.59	0.59	0.19	28.3
-23	0.202	0.051	0.21	0.74	0.72	0.05	10.8
-24	0.198	0.119	0.21	0.70	0.66	0.09	25.5

TABLE V

CALCULATED HOLDUPS, RUN 218--DILUTE PHASE

CATALYST : HDS-2A
GAS : NITROGEN
LIQUID : KEROSENE
COAL CHAR CONC: 0.0 VOL %
TEMPERATURE : 76. DEG F

Run No.	Liquid Flow Rate, Ft/Sec	Gas Flow Rate, Ft/Sec	ELG	ELDP	EGG
218- 1	0.069	0.0	0.99	0.98	0.0
- 2	0.086	0.0	0.99	0.98	0.0
- 3	0.101	0.0	0.98	0.98	0.0
- 4	0.126	0.0	0.99	0.98	0.0
- 5	0.149	0.0	0.99	0.97	0.0
- 6	0.173	0.0	0.99	0.98	0.0
- 7	0.087	0.048	0.91	0.94	0.09
- 8	0.087	0.099	0.82	0.85	0.18
- 9	0.087	0.132	0.76	0.79	0.24
-10	0.087	0.160	0.75	0.78	0.25
-11	0.087	0.193	0.73	0.78	0.27
-12	0.102	0.056	0.90	0.93	0.10
-13	0.102	0.100	0.82	0.84	0.18
-14	0.101	0.143	0.75	0.77	0.25
-15	0.101	0.200	0.71	0.75	0.29
-16	0.151	0.047	0.90	0.92	0.10
-17	0.150	0.108	0.81	0.84	0.19
-18	0.151	0.164	0.75	0.77	0.25
-19	0.172	0.048	0.91	0.92	0.09
-20	0.153	0.223	0.70	0.72	0.30
-21	0.172	0.113	0.83	0.85	0.17
-22	0.172	0.169	0.77	0.78	0.23
-23	0.202	0.051	0.91	0.94	0.09
-24	0.198	0.119	0.84	0.86	0.16

TABLE VI

RICHARDSON-ZAKI PARAMETERS, RUN 218

Method	$\varepsilon_l = \varepsilon_{l\infty}$	$\varepsilon_l = 1 - \varepsilon_c$
Tests	2-6	2-6
n	2.6	3.2
U_t	0.43	0.49

DNR/ml
12/31/80

TABLE VII

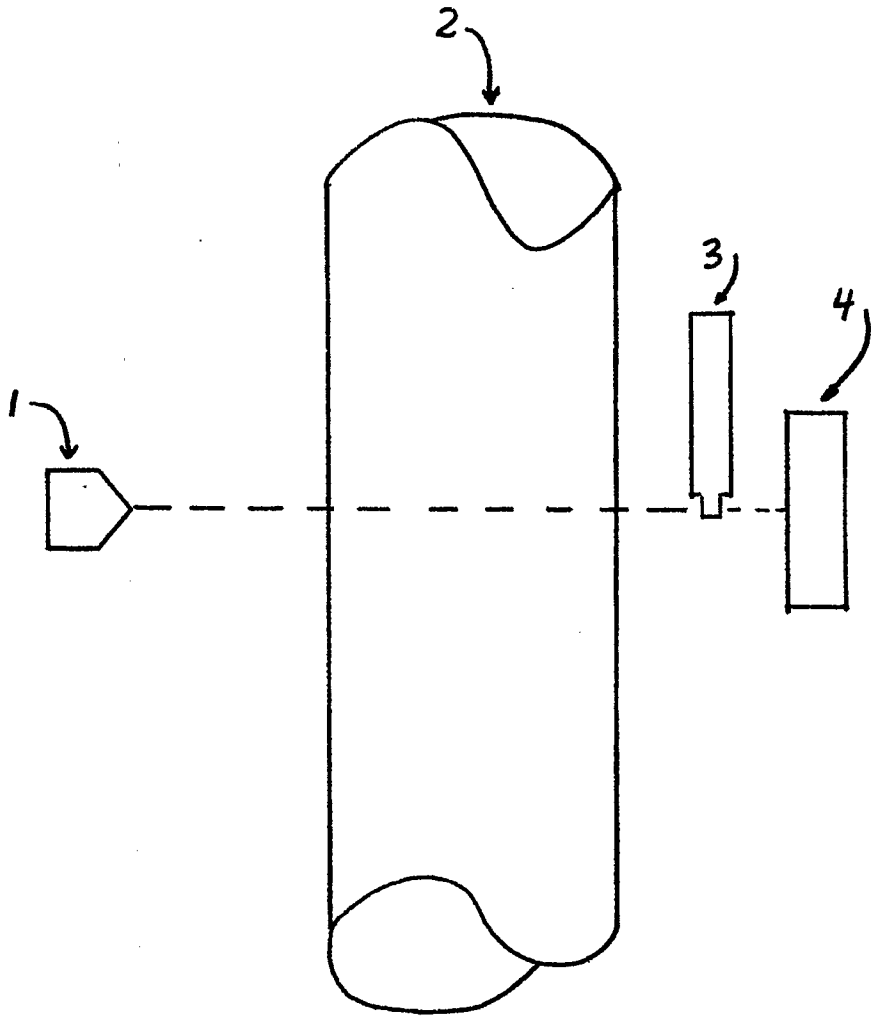
COMPARISON OF RICHARDSON-ZAKI PARAMETERS

Run	Date	n	U_t
201	8/30/78	2.68	0.48
212	4/12/79	3.58	0.45
218	5/29/80	2.6	0.43

DNR/ml
12/31/80

Figure 1

SIDE VIEW OF DENSITY GAUGE ASSEMBLY

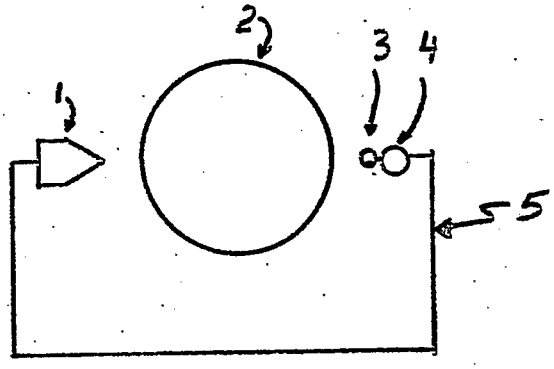


Legend

1 500 mc Cs-137 Source
2 PDU Reactor
3 Amoco Scintillation Detector
4 HRI Geiger/Muller Detector

Figure 2

TOP VIEW OF DENSITY GAUGE ASSEMBLY

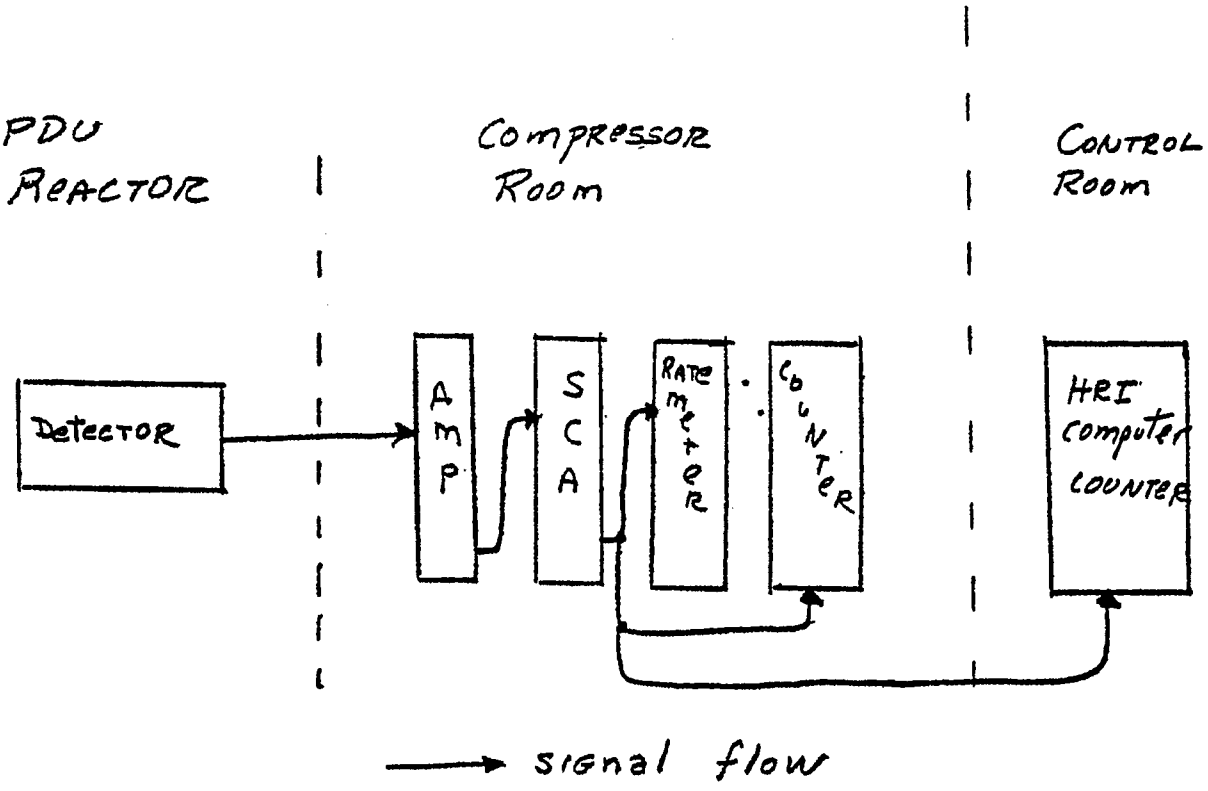


Legend

1 500 mc Cs-137 Source
2 PDU Reactor
3 Amoco Scintillation Detector
4 HRI Geiger/Muller Detector
5 Traverse Sled Assembly

Figure 3

AMOCO DETECTION SYSTEM SCHEMATIC



Notes

The SCA puts out a ⎍ pulse if the signal from the amplifier is in the proper energy range. One pulse corresponds to one detected gamma-ray photon.

The rate meter is an analog indicator of the SCA pulse rate (counts/sec).

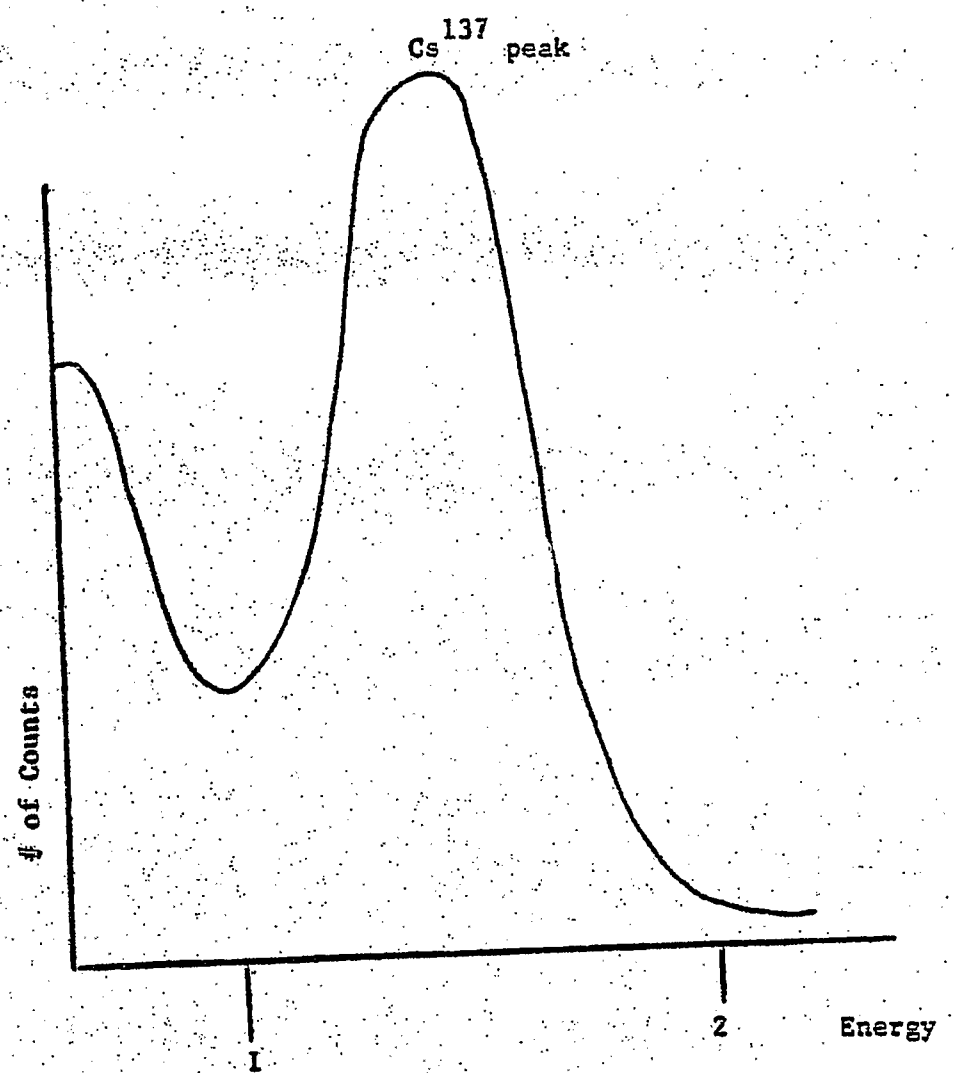
The Amoco counter (there are two available) counts the SCA pulses digitally over a preset time period.

The HRI counter is attached to the same SCA output as the Amoco counter.

The Amoco counter and rate meter always appear consistent with each other. Both available Amoco counters agree with each other.

Figure 4

ENERGY WINDOW CRITERIA



1. Lower edge: Minimum between background and Cs-137 peak.
2. Upper edge: Defined as the energy where count rate = 10% of maximum rate.

Figure 5

APPARENT SHIFT OF Cs-137 PHOTOPEAK WITH TEMPERATURE

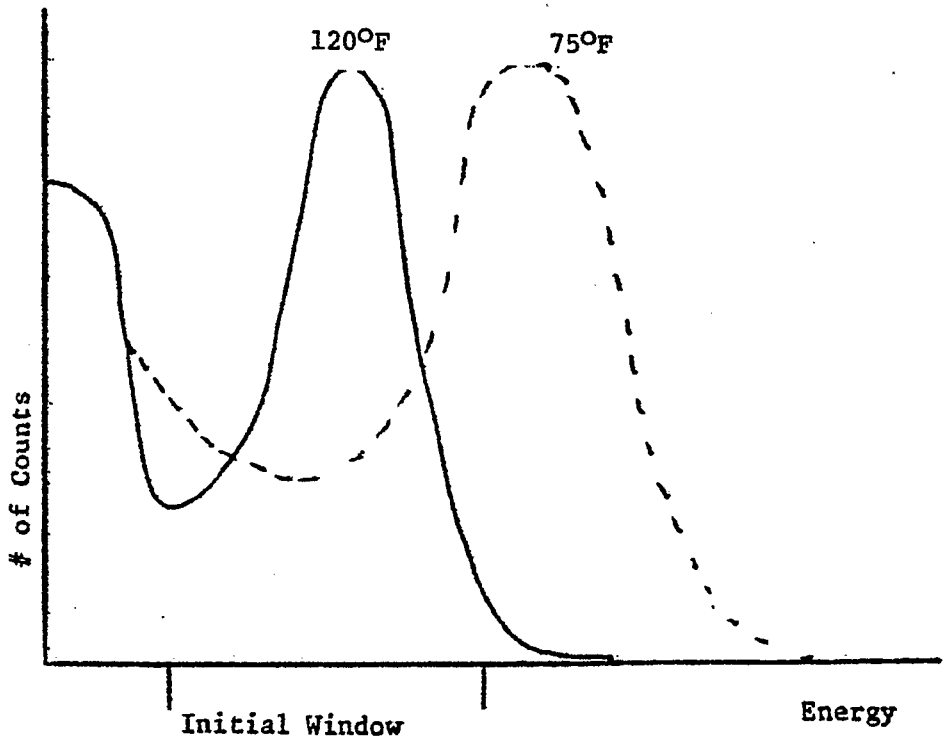


Figure 6

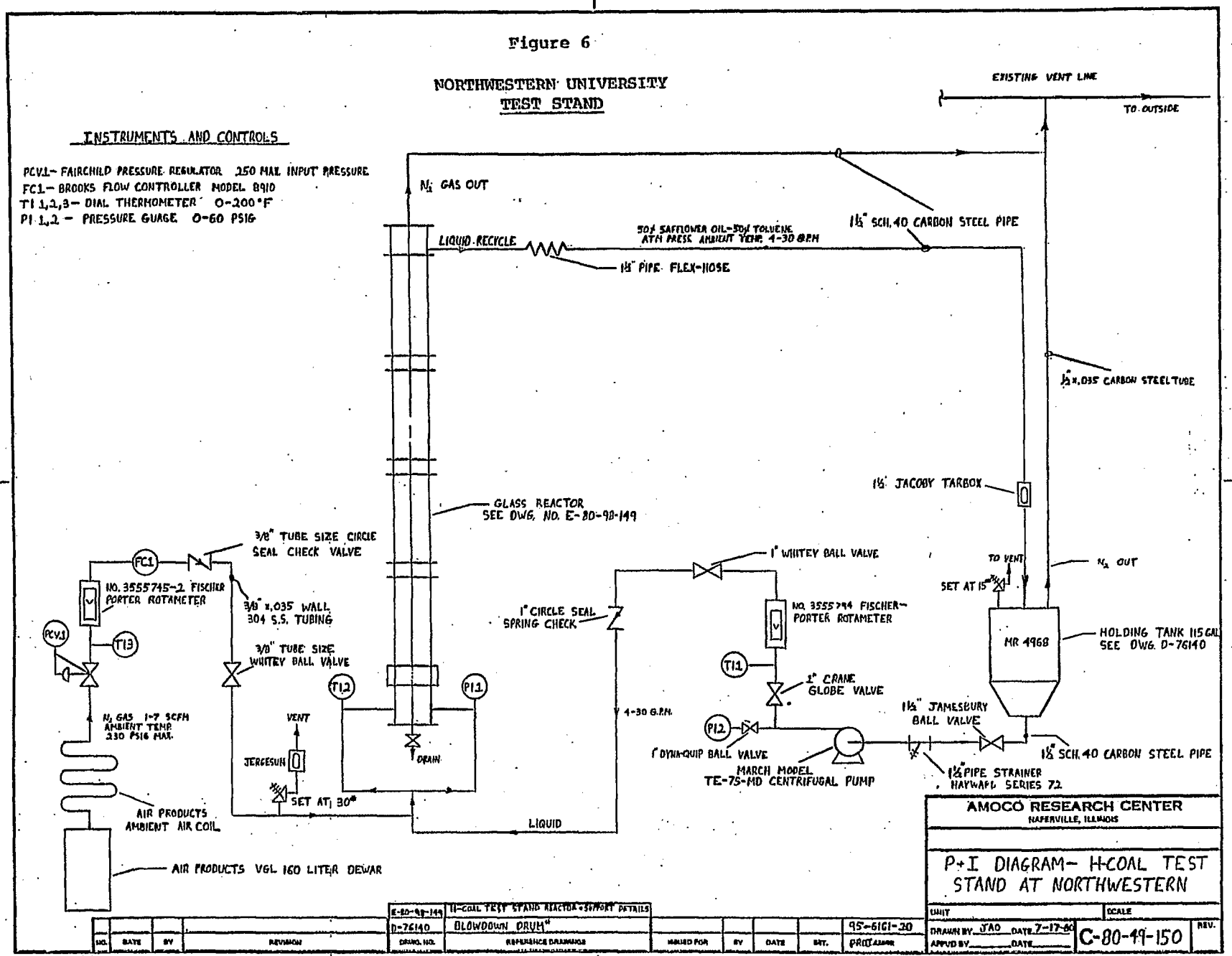

NORTHWESTERN UNIVERSITY
TEST STAND
INSTRUMENTS AND CONTROLS
PCV1- FAIRCHILD PRESSURE REGULATOR 250 MAX INPUT PRESSURE
FC1- BROOKS FLOW CONTROLLER MODEL 8910
TI 1,2,3- DIAL THERMOMETER 0-200°F
PI 1,2 - PRESSURE GUAGE 0-60 PSIG
EXISTING VENT LINE
TO OUTSIDE
N2 GAS OUT
LIQUID RECYCLE
50% SAFFLOWER OIL-50% TOLUENE ATM PRESS AMBIENT TEMP 1-30 G.P.M.
1½" SCH.40 CARBON STEEL PIPE
1½" PIPE FLEX-HOSE
½" x.035 CARBON STEEL TUBE
1½" JACOBY TARBOX
GLASS REACTOR SEE DWG. NO. E-80-98-149
3/8" TUBE SIZE CIRCLE SEAL CHECK VALVE
FC1
NO. 3555745-2 FISCHER PORTER ROTAMETER
3/8" x.035 WALL 304 S.S. TUBING
PCV1
TI3
3/8" TUBE SIZE WHITEY BALL VALVE
TI2
PI1
N2 GAS 1-7 SCFH AMBIENT TEMP 230 PSIG MAX.
VENT
JERGESON
SET AT 30#
DRAIN
AIR PRODUCTS AMBIENT AIR COIL
AIR PRODUCTS VGL 160 LITER DEWAR
LIQUID
1" CIRCLE SEAL SPRING CHECK
1" WHITEY BALL VALVE
NO. 3555794 FISCHER-PORTER ROTAMETER
TI1
1" CRANE GLOBE VALVE
1-30 G.P.M.
PI2
1" DYN-QUIP BALL VALVE
MARCH MODEL TE-7S-MD CENTRIFUGAL PUMP
TO VENT
SET AT 15#
N2 OUT
MR 4968
HOLDING TANK 115 GAL SEE DWG. D-76140
1½" JAMESBURY BALL VALVE
1½" SCH.40 CARBON STEEL PIPE
1½" PIPE STRAINER HAYWARD SERIES 72
AMOCO RESEARCH CENTER
NAPERVILLE, ILLINOIS
P+I DIAGRAM- H-COAL TEST STAND AT NORTHWESTERN
UNIT
SCALE
DRAWN BY JAO DATE 7-17-80
APPVD BY DATE
C-80-49-150
REV.
E-80-98-149 H-COAL TEST STAND REACTOR + SUPPORT DETAILS
D-76140 BLOWDOWN DRUM
95-6161-20
NO. DATE BY REVISION
DRWG. NO. REFERENCE DRAWINGS
ISSUED FOR BY DATE SHT. PRINTARM

Figure 7

ZERO GAMMA-RAY SCAN OF PDU REACTOR

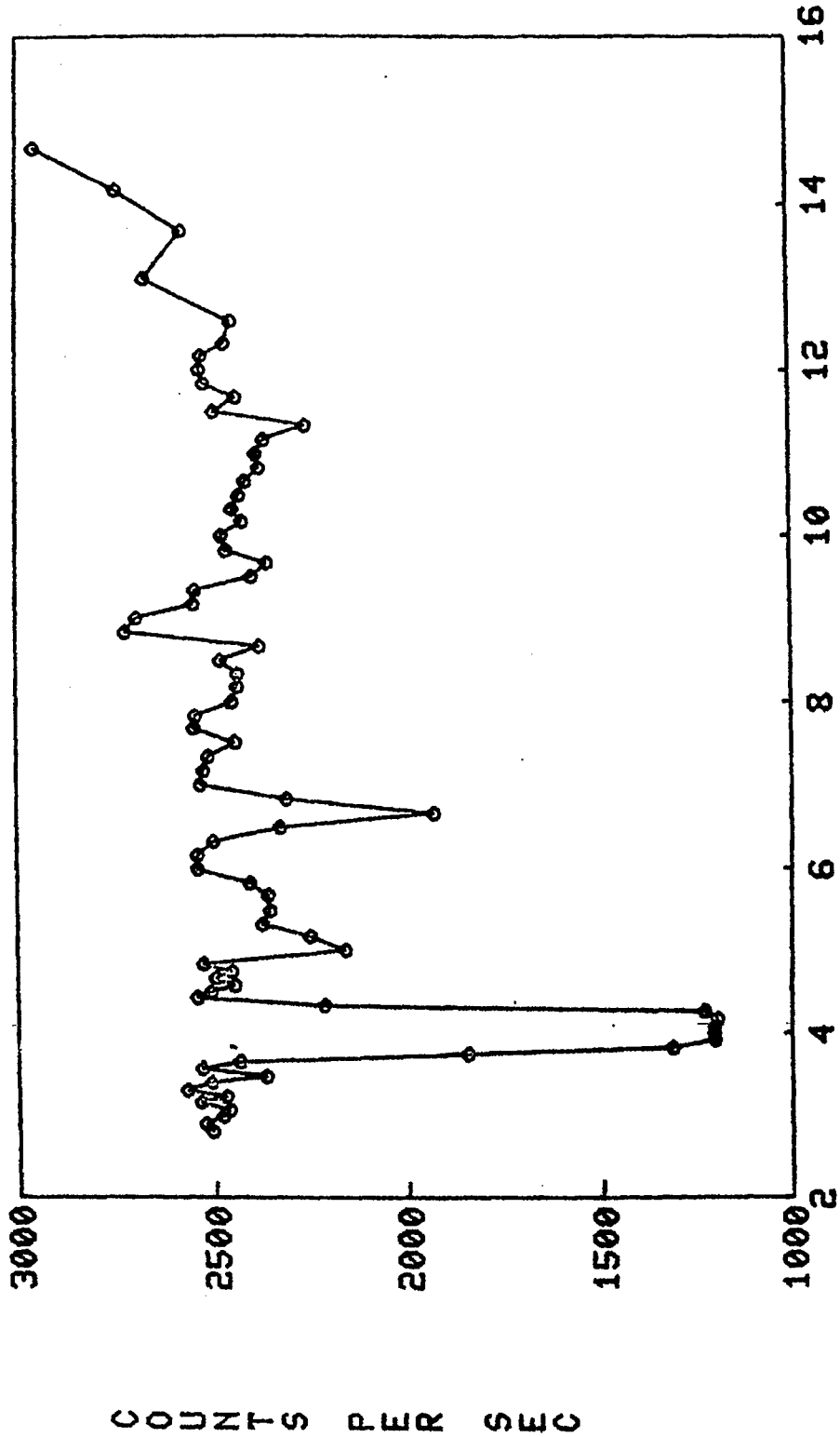


Figure 8

GAMMA-RAY SCAN OF REACTOR, 7/30/80 AND 8/5/80

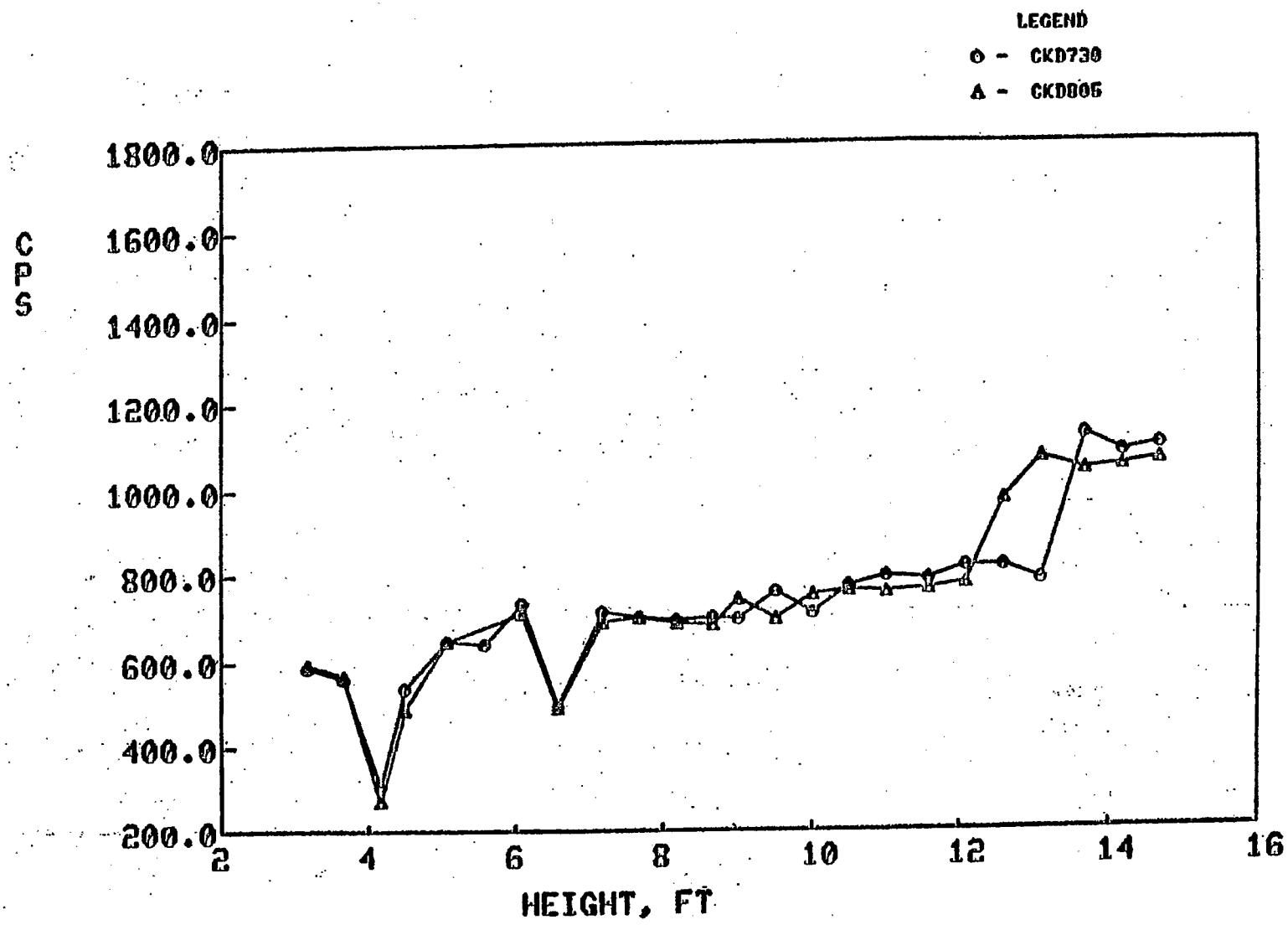


Figure 9

GAMMA-RAY SCAN OF REACTOR, 9/4/80 AND 9/11/80

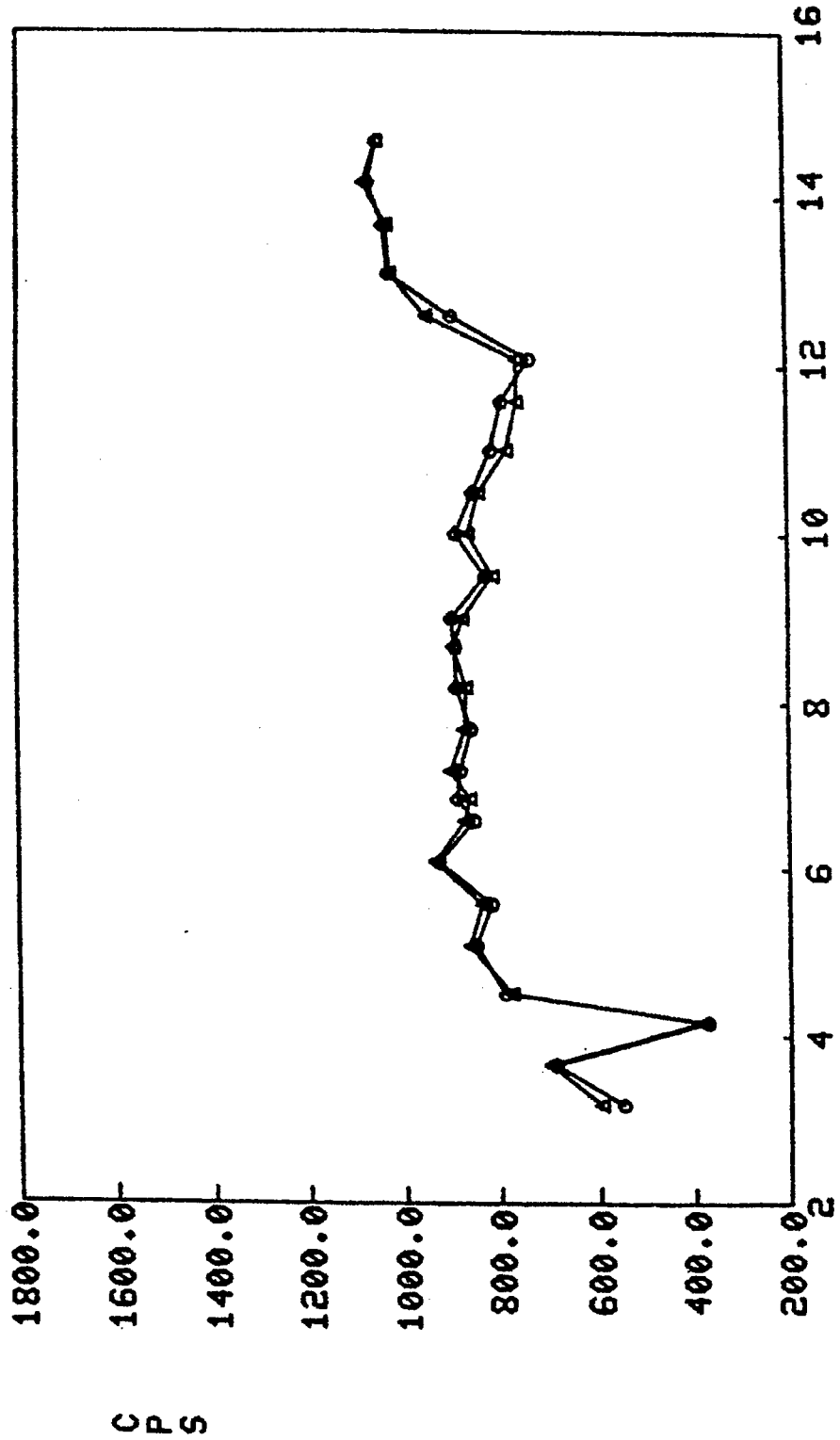


Figure 10

GAMMA-RAY SCAN OF REACTOR, 9/19/80 AM AND PM

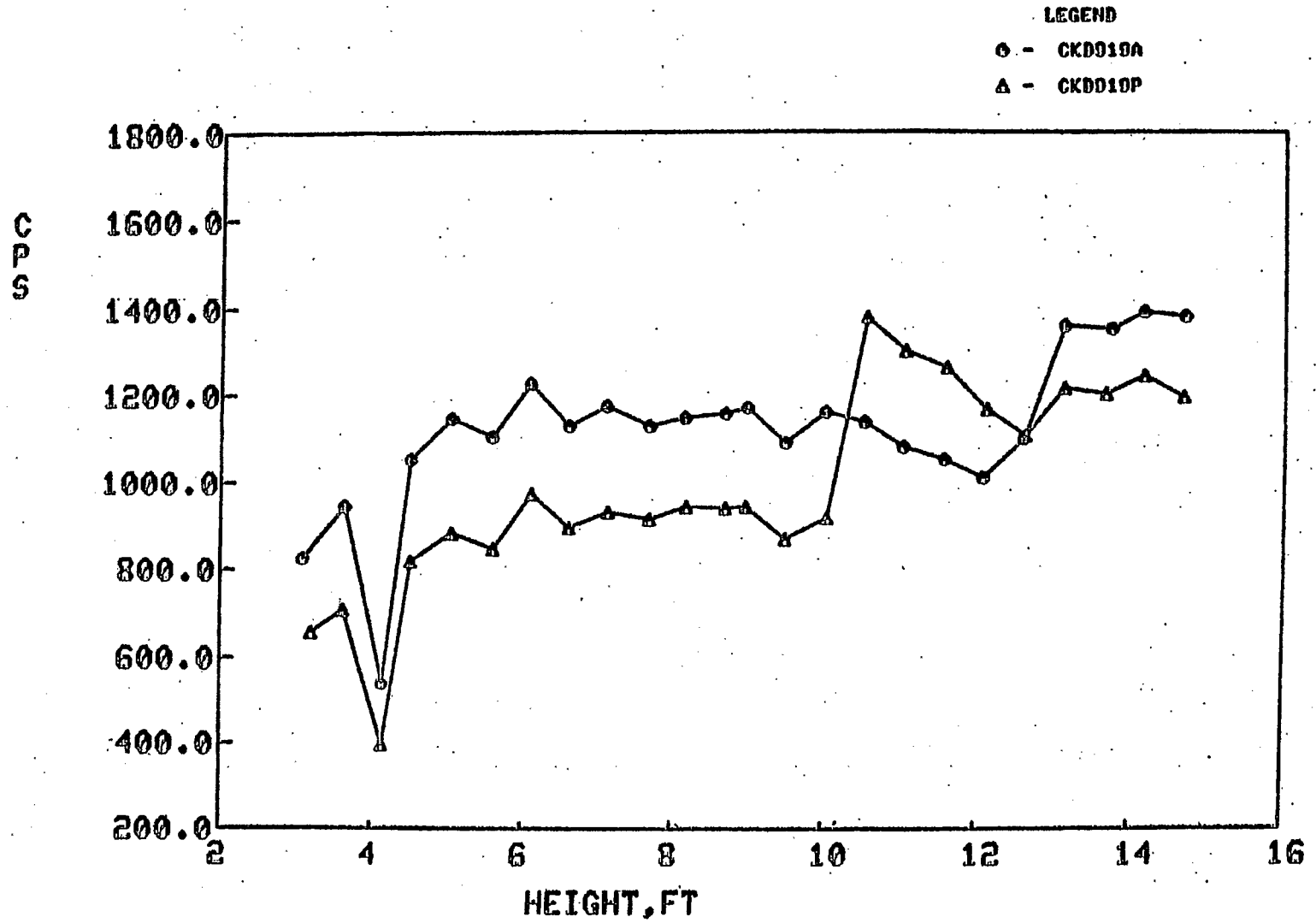


Figure 11

GAMMA-RAY SCAN OF REACTOR, 9/20/80 AM AND PM

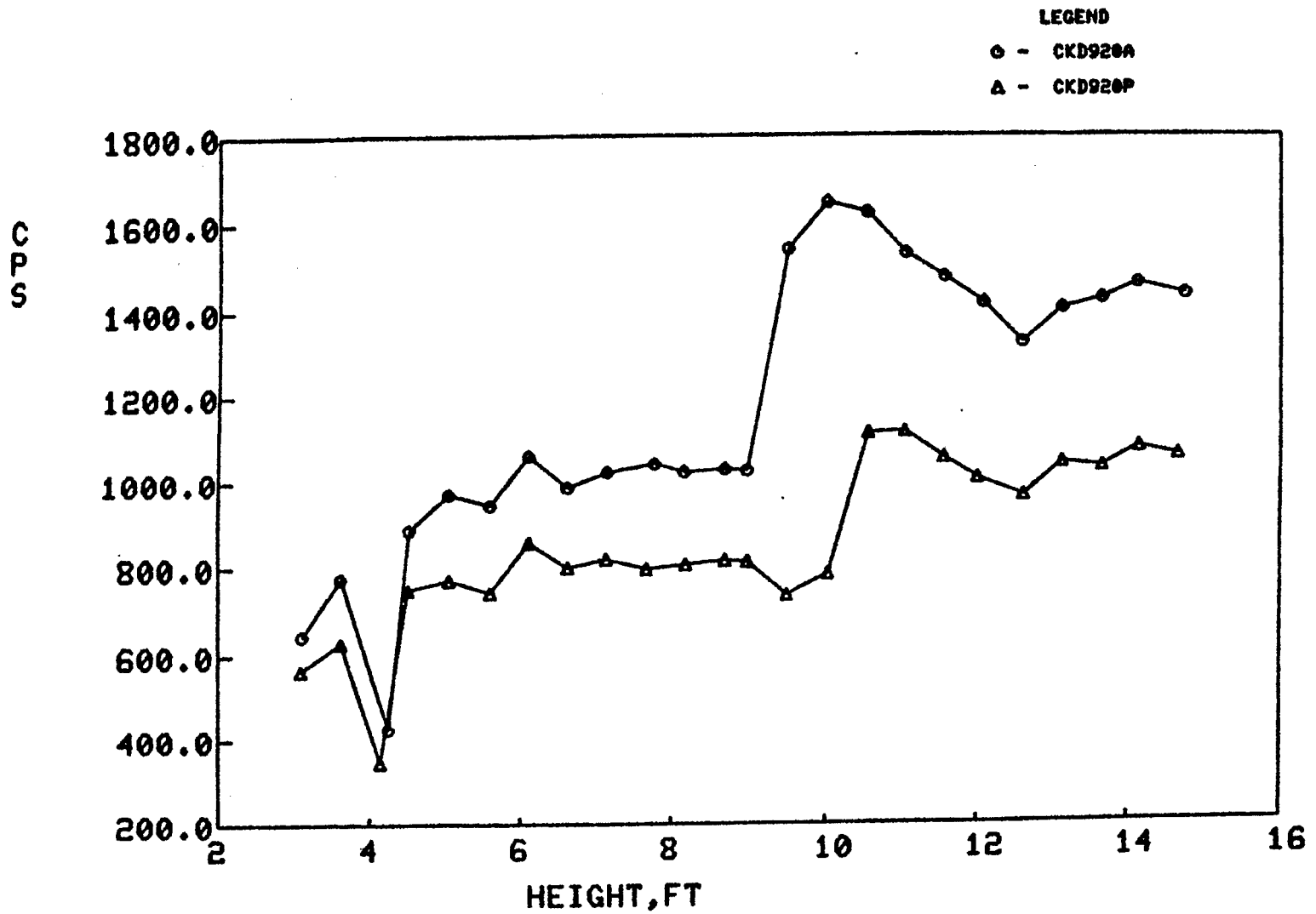


Figure 12

GAMMA-RAY SCAN OF REACTOR, 9/21/80 AM AND PM

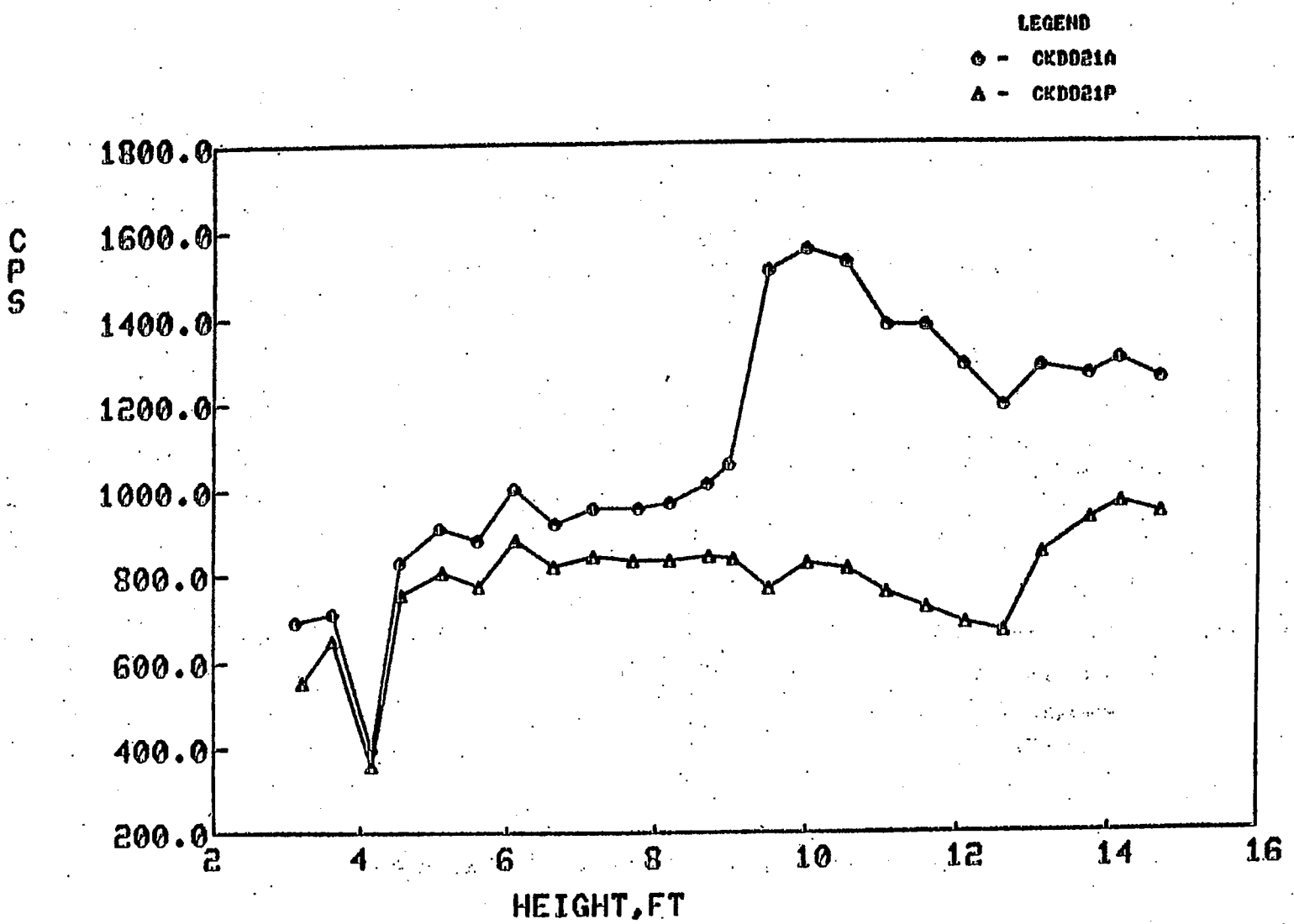


Figure 13

GAMMA-RAY SCAN OF REACTOR, 9/22/80 AM AND PM

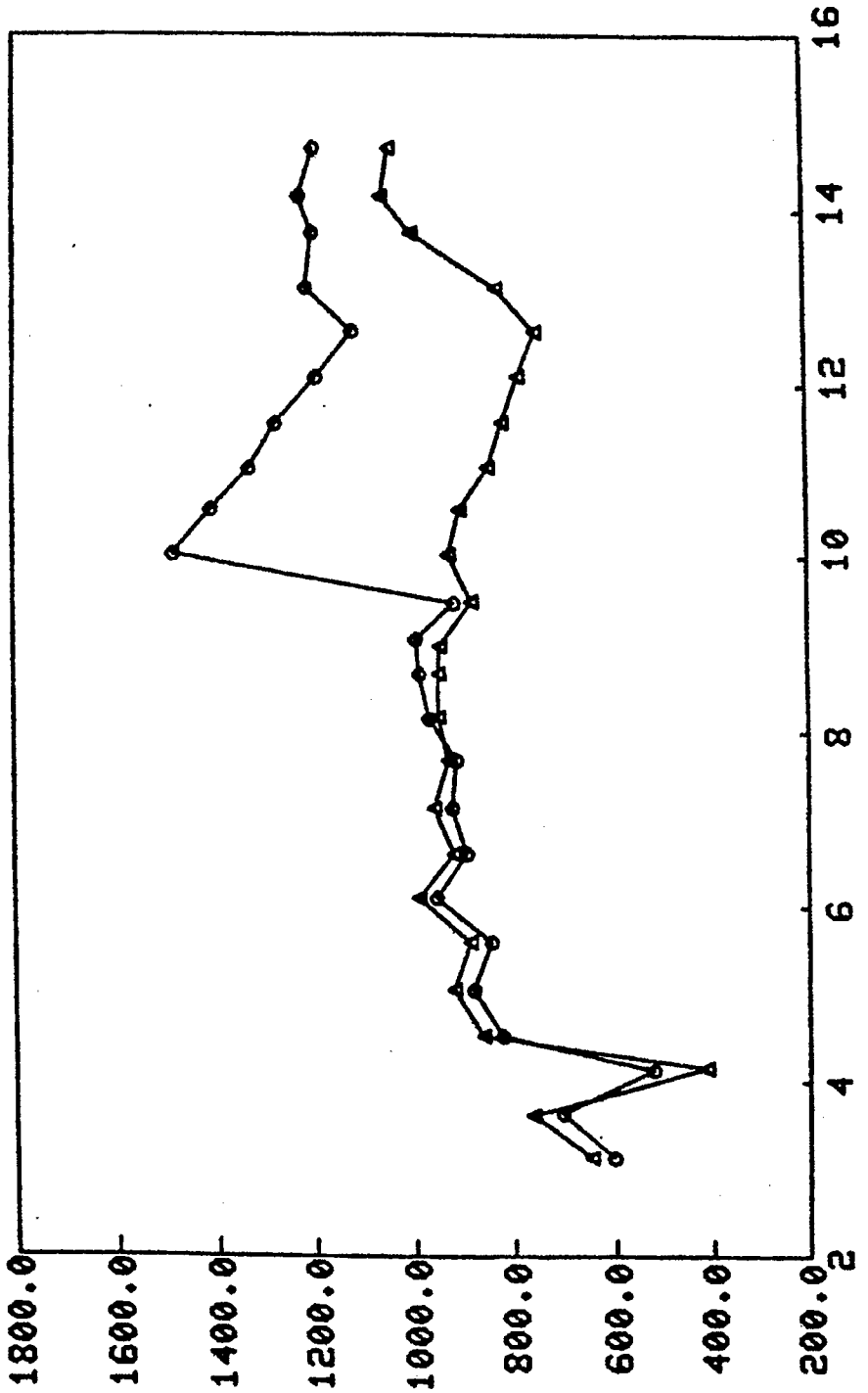


Figure 14

GAMMA-RAY SCAN OF REACTOR, 9/23/80 AM AND PM

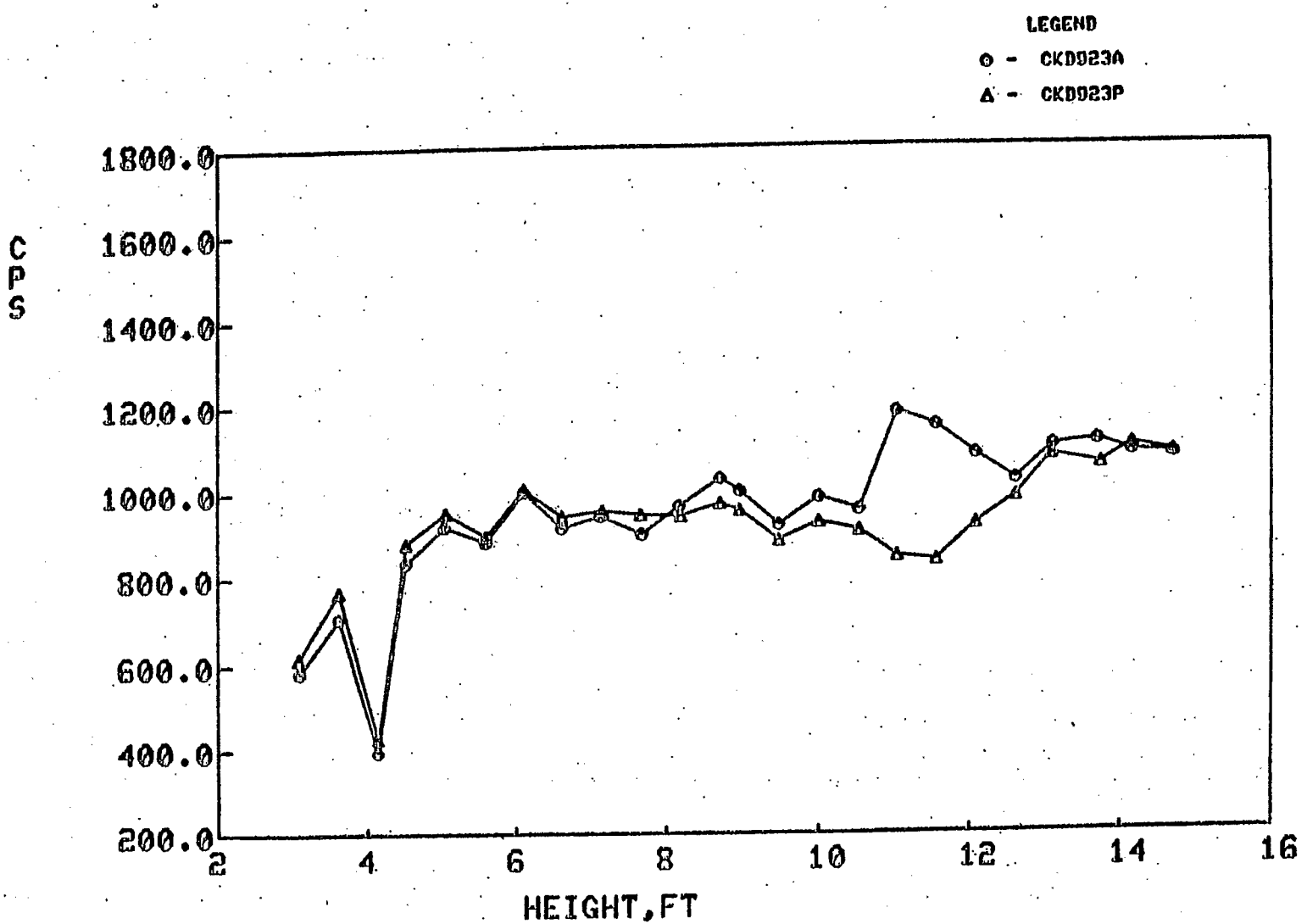


Figure 15

GAMMA-RAY SCAN OF REACTOR DURING SHUTDOWN, 9/24/80 12:26 PM (A) AND 7:06 PM (P)

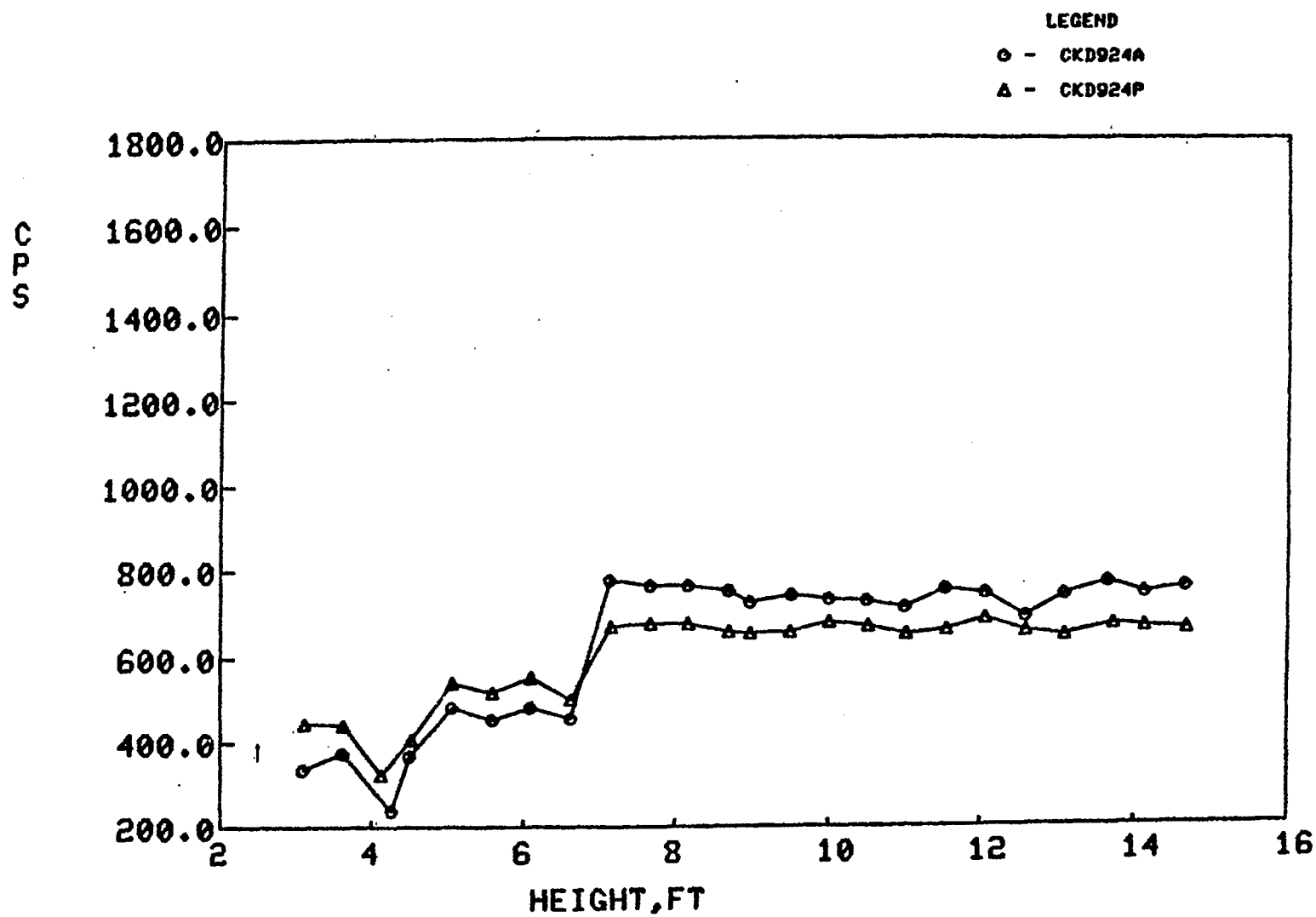


Figure 16

BED VOID FRACTION VS. LIQUID VELOCITY

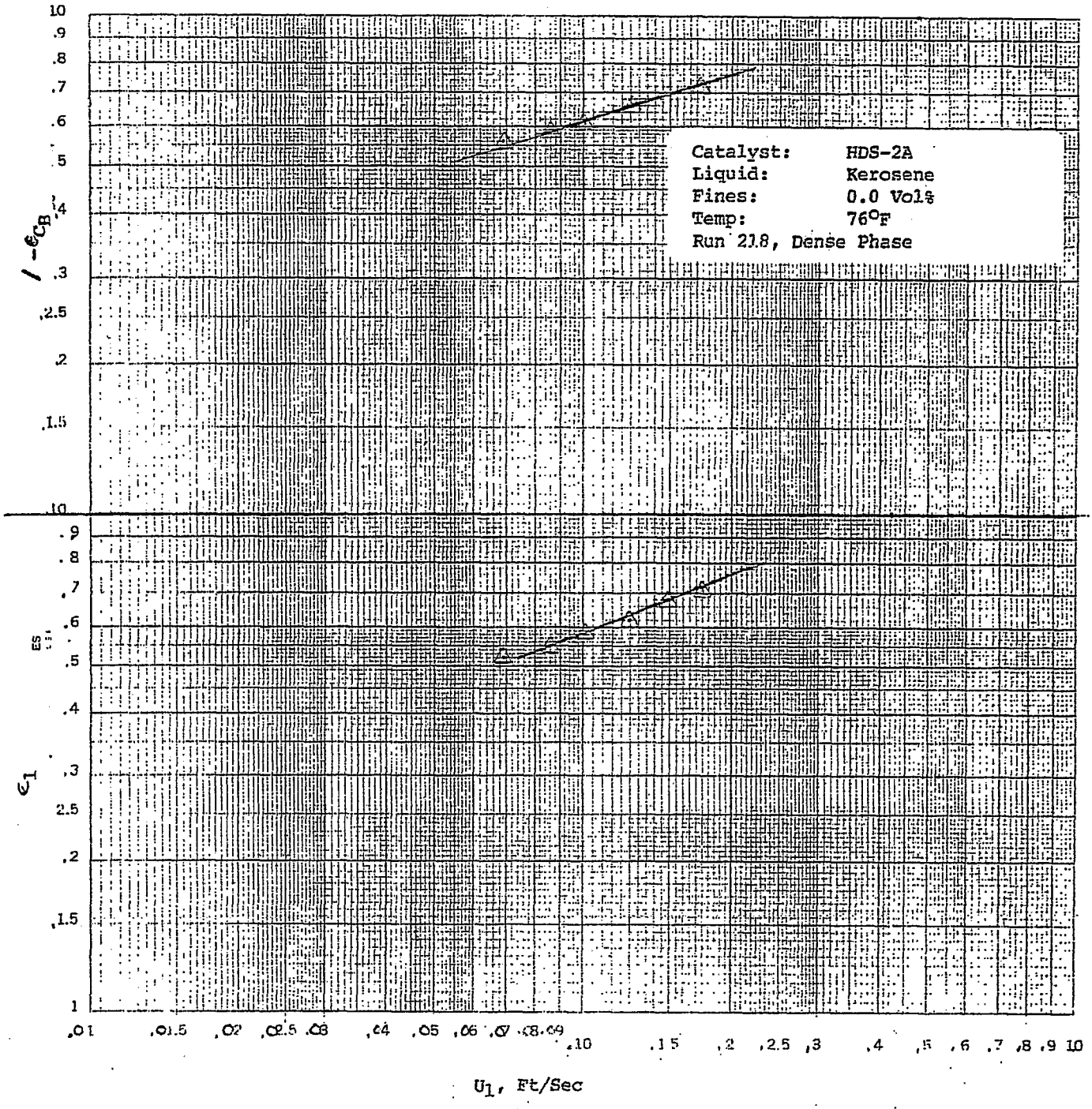
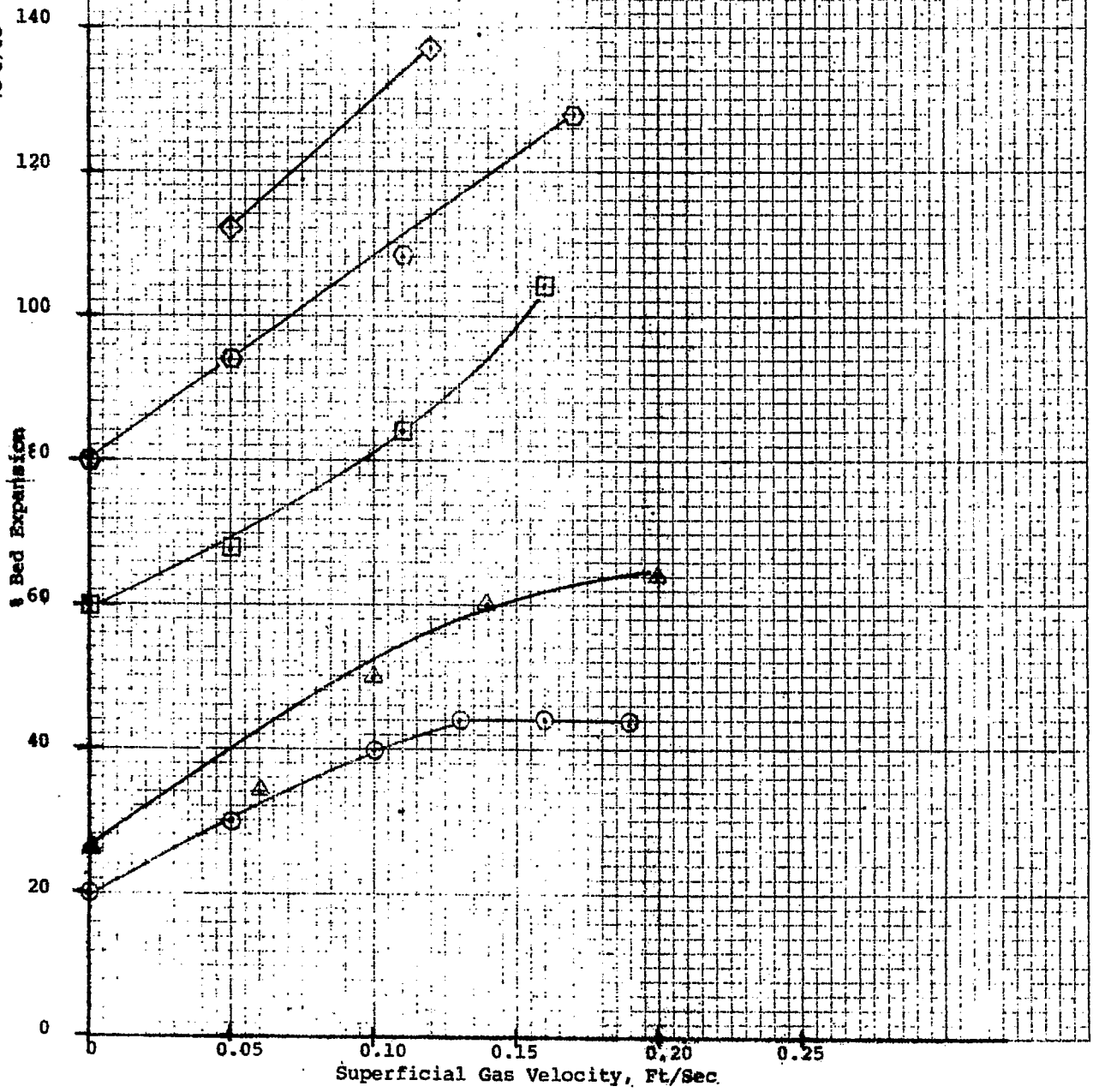


Figure 17

CATALYST BED EXPANSION

		Legend	U_1, Ft/Sec
Run:	218		0.09
Liquid:	Kerosene		0.10
Gas:	Nitrogen		0.15
Catalyst:	HDS-2A (1/16 x 3/16")		0.17
Fines Conc:	0.0 Vol%		0.20



46 0703

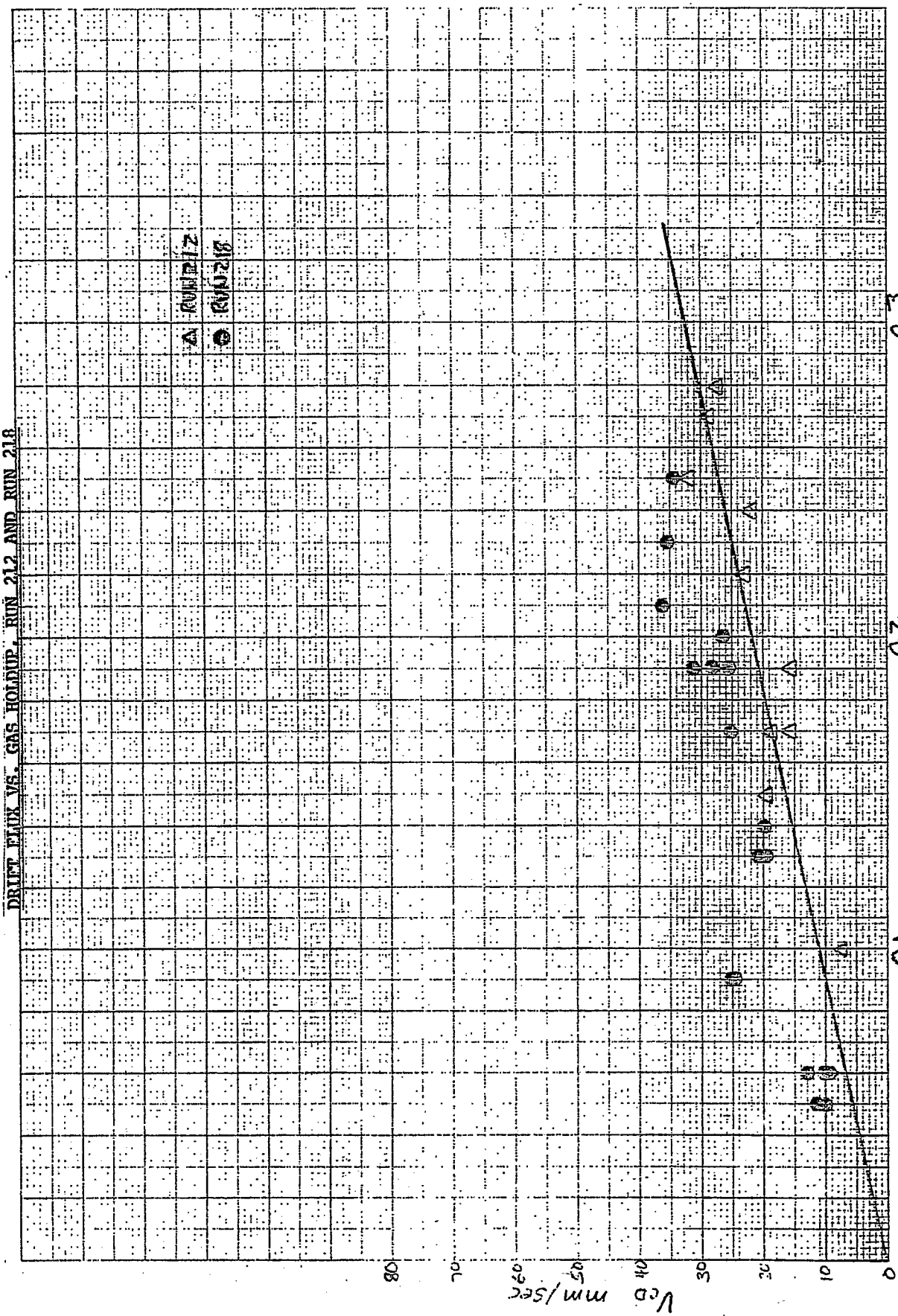


Figure 18

46 1320

APPENDIX A

PROCEDURE FOR TAKING VISCOSITY SAMPLES

PROCEDURE FOR TAKING VISCOSITY SAMPLES

1) Check equipment to be sure it is assembled correctly as per attached drawing. Be sure to check electrical tapes and temperature controls.

2) Connect unit to PDU-130 sampling point, (Near Edwards let-down valve) from high pressure separator.

3) Purge system with nitrogen

   a) At start of purge have all valves open except for Edwards let-down valve.

   b) Check for nitrogen flow at the 4 valves open to the atmosphere.

   c) After about a minute of purge close the valves open to atmosphere.

   d) Raise nitrogen pressure to about 2000 psig.

   e) Close valve leading to regulator. (V13)

   f) Raise system temperature to 350°F.

   g) Check system for nitrogen leaks.

   h) Crack purges to reduce system pressure to (about) 100 psig. Be sure to purge nitrogen while reducing pressure.

   i) Close all purge valves (V12, V2, V3, V9, V10, V11)

   j) Raise system temperature to 350°F and then close valve to pressure gauge. (V14)

   k) Open valves to sample and purge bombs so they can be filled.

   l) Open Edwards (V4) letdown valve to fill. Sample and purge bomb.

   m) After temperature in sample bomb rises sharply, close valves (V5, V6) on both sides of the sample bomb and turn off electrical tape on sample bomb.

n) Close Edwards relief valve. (V1)
OPEN V2, V3, and V13

o) Purge the line between the valve before the sample bomb and the Edwards relief valve with 100-200 psi nitrogen.

p) Close valves in feed line.
OPEN Valves V11, V12 Before Removing Sample Bomb

q) After temperature in sample bomb is lowered, remove it, attach capitance bomb, double valve sample bomb top, and place in storage.

r) Put new sample bomb in place.

s) Purge purge bomb with 100-200 psi nitrogen via new sample bomb.
OR THROUGH V11

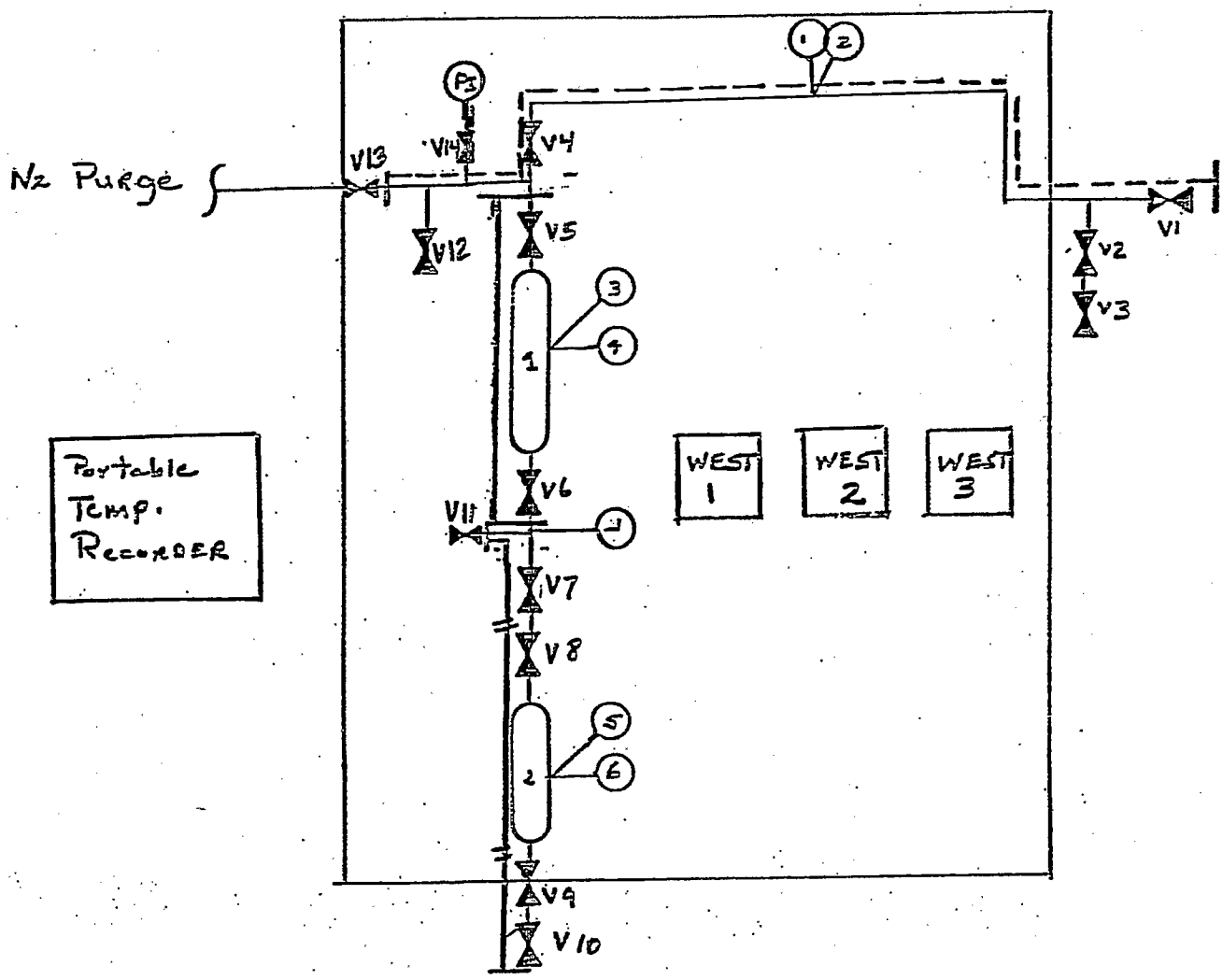
t) Close all remaining open valves.

u) Reduce pressure on nitrogen regulator to 100 psig.

v) Turn off electrical tapes.

w) Wrap new sample bomb with electrical tape.

1 Skin control of Winding
2 Skin E to Recorder.
3 Skin control of Winding
4. Skin E to Recorder.
5. Skin control of Winding
6 Skin E to Recorder
7 Internal E for Confirming Flow.



Note.

1 Bomb-1 Sample Bomb
2 Bomb-2 Purge Bomb.
3 All Tubing 9/16 X 5/16 AE 316 SS.
4. All Valves 9/16 Med. Press 316 SS
5.

ET-1
ET-2
ET-3

4-10-80
D.T

APPENDIX B

NORTHWESTERN UNIVERSITY PROGRESS REPORT

APPENDIX B

PROGRESS REPORT ON AMOCO DOE CONTRACT

"ON H-COAL FLUID DYNAMICS"

In July and August, we concentrated on two tasks:

(1) Selection of a mixture fluid having the correct fluid properties, $\rho \simeq 1$ gm/cc, $\mu \simeq 1$ to 4 c.p. and η (refractive index) $\simeq 1.5$, so that it matches the refractive index of the catalyst simulator material.

The result of the investigation shows that the best combination is DPM - 66% plus Diphenyl Ether - 34% with the following physical properties,

$\eta = 1.474$

$\mu = 3.5$ cp

$\rho = .99$ gm/cc

σ = (surface tension) = 29.4 dynes/cm

A summary of the investigation is shown as Appendix A.

(2) The experimental apparatus was delivered to N.U. in the first week of August. Some piping was changed to accommodate the optics. The pyrex glass rods were ordered and a jig was set up to cut the 2 mm rod to about 5 mm length. The optical table and layout were also designed.

In September, the major accomplishments were:

(1) The optical table was built and installed. It can traverse the entire vertical length ($\sim$ 12 ft).

(2) The LDV system is being built. All the necessary optical parts have been ordered and most of them have arrived. The same is true for the holographic optical system.

(3) The DPM fluid was installed and recirculated in the system. At higher flow rate (15 gal/min) it was found that gas was sucked from the storage tank on the suction side of the pump which causes flow fluctuations. The higher flow rate also causes flow blockage at the gas exhaust vent at the top of the column.

We plan to install a vane at the exit of the storage tank to prevent swirling and it should alleviate the problem of gas entrainment into the piping system. The return pipe is now at a steep angle to facilitate the flow of fluid from the overflow weir at the top of the column. The gas vent is being first connected to the storage tank and then venting out into the atmosphere.

APPENDIX A

Selection of Fluids and Catalyst Simulator Material for an Optically Clear Fluidized Bed.

Numerous possibilities were considered in searching for a clear fluid and a clear solid with the same refractive index, correct fluid viscosity, and proper relative densities. The initial area of search centered around finding a fluid compatible with some clear plastic. The possible plastics (such as Plexiglas or Lexan) all tend to interact with nearly any fluid they are immersed in to at least some small extent. For example, Plexiglas increases in weight by about 0.5% when left in water for seven days at room temperature.[1] Since it will be necessary to match the refractive index to within at least 10^{-3},[2] any time dependent interaction between the fluid and solid is very undesirable. Consequently it was decided to use glass instead of plastic, and the most readily available glass was Pyrex 7740 with

Refractive Index $n_d = 1.474$

Density $\rho = 2.23$ gm/cc

The problem was thus reduced to finding a realtively safe fluid with the proper physical properties.

Refractive Index $n_d = 1.474$

Viscosity $\mu = 1.0 - 4.0$ cp

In addition, because the "fluid" would actually have to be a combination of fluids, the vapor pressure of the most volatile component would have to be low enough so that its evaporation over a several hour period would not effect the refractive index match. Since gas will be bubbling through the system and carrying away essentially saturated vapor from the fluid, this eliminates any

fluid with a vapor pressure above approximately 5 mm Hg.

A list of possible fluid combinations was assembled considering both safety and physical properties, and the top seven combinations are given in Ref. 3. Small samples of the necessary fluids for the top two combinations were obtained to verify their miscibility in the proper proportions. The two combinations are

1) Dipropylene Glycol Moromethyl Ether (Dowanol DPM) 66%, plus Diphenyl Ether - 34%

   Refractive Index: $n_d = 1.474$

   Viscosity: $\mu = 3.5$ cp

   Density: $\rho = 0.99$ gm/mℓ

   Surface Tension: $\sigma = 33.4$ dynes/cm (calc.)

   $= 29.5$ dynes/cm (exp.)

   Also vapor pressure: Dowanol DPM $\simeq 0.4$ mm Hg

   Diphenyl Ether $\simeq 0.02$ mm Hg

2) Dowanol DPM - 43%, plus Dimethyl Phthalate - 57%

   Refractive Index: $n_d = 1.474$

   Viscosity: $\mu = 11.2$ cp

   Density: $\rho = 1.09$ gm/mℓ

   Surface Tension: $\sigma = 40.3$ dynes/cm

   And vapor pressure: Dimethyl Phthalate < 0.01 mm Hg

In order to better match the surface tension values with actual H-Coal process numbers ($\sigma \simeq 3$ to 9 dynes/cm), it is quite likely that the addition of a small amount of a proper chemical would reduce the stated values to the desired range.

For initial experimental work, we will be using the Dowanol DPM plus Diphenyl Ether combination.

1. Modern Plastics Encyclopedia, 1977-1978, p. 505

2. C. P. Wang, J. B. Bernard, and R. H. Lee, "Feasibility of Velocity Field Measurement in a Fluidized Bed with a Laser Anemometer." Presented at the Third International Workshop on Laser Velocimetry, July 11-13, 1978.

3. Letter from Paul Meernik to Dr. Robert Schaefer, July 15, 1980.

*U.S. GOVERNMENT PRINTING OFFICE: 1980-740-145/1092