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From C. JND

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NOTICE

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SOCONY MOBIL OIL COMPANY, INC.

RESEARCH DEPARTMENT

MONTHLY PROGRESS MEMORANDUM
(Covering December 16, 1964 to January 15, 1965)

ANVIL POINTS OIL SHALE RESEARCH CENTER

Rifle, Colorado

January 22, 1965

CONTRIBUTORS:

Mechanical Engineering
Retorting Section
Analytical Laboratory Section
Engineering and Economic Analyses

Signed by:

R. H. Cramer
Program Manager

NOTICE

The primary objective of the Anvil Points Oil Shale Research Center MONTHLY PROGRESS MEMORANDUM is to advise authorized personnel employed by the Participating Parties[1] that various activities are in progress or that certain significant data have been obtained within the Research Center.

These MONTHLY PROGRESS MEMORANDA have been prepared to provide rapid, on-the-spot reporting of research currently in progress at Anvil Points. The conclusions drawn by project personnel are tentative and may be subject to change as work progresses. The PROGRESS MEMORANDA have not been edited in detail.

[1] Socony Mobil Oil Company, Inc., Project Manager

Humble Oil and Refining Company

Continental Oil Company
Pan American Petroleum Corporation
Phillips Petroleum Company
Sinclair Research, Inc.

MONTHLY PROGRESS MEMORANDUM

(Covering December 16, 1964 to January 15, 1965)

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CONFIDENTIAL

MONTHLY PROGRESS MEMORANDUM
(Covering December 16, 1964 to January 1965)

I. TECHNICAL ADVISORY COMMITTEE

The fourth meeting of the Technical Advisory Committee was held on January 8, 1965 at the Humble offices in Tulsa, Oklahoma. Representatives from each of the Participating Parties attended.

Each of the Participating Parties will send Technical Observers and/or Committee Members to the Research Center during the week of January 18 for detailed technical discussions with members of the staff.

The next meeting of the Committee will be held on March 11, 1965 at the Research Center. At this time, a revised 1965 budget will be presented which will take into account the $127,000 underrun during 1964 and other factors which have changed since the first 1965 budget was prepared last August.

II. MECHANICAL ENGINEERING (W. S. Bergen)

Initial test runs of the Retort No. 2 were completed during this period. Reconstruction of and revisions to the Retort No. 1 and its recovery system were also completed. The Retort No. 1 went back on stream December 26.

Shale drying needs were studied and equipment installed to alleviate handling and operating problems with wet shale.

Summary

1. Retort No. 2

The retort was shut down at the conclusion of the second 9-day operating period. Test runs during this two week interval incorporated the use of the electrostatic precipitator for better recovery, the Syntron shale feed integrator for better material balances, equipment to continuously sample retort gas and located in the analytical laboratory, traps for fines in the liquid recovery system, and redesigned shale flow chutes to prevent bridging and to provide more disengaging area at the top of the retort.

All changes proved beneficial.

Many problems still remain to be solved.

2. Retort No. 1

All planned revisions were completed prior to putting the unit back on stream. Included in the revisions were a new center section for the retort to replace the warped portion, a modified air-gas distributor design, a surge drum at the retort top outlet to disengage shale fines ahead of the recovery system, an air cooled condenser and accumulator after the demister, an additional recycle gas entry into the center of the bed just above the table feeder, and repiping of the electrostatic precipitator.

All new features were tested. Most proved effective.

Wet shale problems continue to plague the operation of the unit due to freezing and mudding of belts and chutes.

3. Shale Drying

The design and drafting of shale drying equipment for 60 ton bins was completed. Construction has started.

Equipment needed to dry shale in the 100 ton bins was also installed and put into service January 12. Priority was given to this project to insure dry shale for immediate operation. This drying operation is proving successful.

4. Mechanical Model

Construction of piping to the air-gas distributor model was completed and tested. Due to the lack of visible smoke generated with the smaller smoke bombs, larger generator holders were designed. These will accommodate smoke bombs with greater capacities.

Discussion

A. Retort No. 2

Modifications were made to this unit during the four day shutdown to improve product yield, recovery, and unit operability.

Shale bridging in the retort standpipe and in the surge hopper above the Syntron feeder was completely eliminated. The standpipe modification was also made to develop a larger disengaging area at the top of the shale bed in retort. As reported earlier the standpipe exit was changed from a 10" X 30" section to a 14" X 15" outlet. The effect of the greater disengaging area is discussed in the Retort Section report.

The electrostatic precipitator was piped into the recovery train for this operating period. This unit is not sized to handle the amount of gas recirculated without a loss in its operating

efficiency. Excessive pressure drops also developed. However, a considerable amount of product was recovered in this recovery unit. The Retort Section has reported on its effectiveness with power both on and off.

Fines in the product oil - a result of both dust on the shale and high fines in the shale charge - had plugged liquid lines during the first 10-day operating period. A fines trap, in the form of an enlarged decanter, was installed under the low pressure cyclone and the demister. The decanter consisted of a four inch pipe, 24 inches long. This proved effective.

The sheave speed on the recycle compressor was changed to lower its capacity by 10%. This change was made to decrease the heating effect of the bypass gas as it mixes with the retort off-gas.

Wet shale, fines laden shale, and shale segregation are problems that remain to be solved. There are definite indications that these factors affected the product make. These factors are discussed in the Retort Section report.

Future plans for the Retort No. 2 include enlargement of the upper portion of the retort, incorporation of a surge drum to settle fines ahead of the low pressure cyclone, and other changes deemed necessary to improve the retorting efficiency.

B. Retort No. 1

Extensive revisions were made in the Retort No. 1. The air-gas distributor was changed when the new center section was installed. New peripheral tubes of 1/4 inch Sch. 40 pipe replaced the original 3/8 inch Sch. 40 pipe. These tubes now extend one inch into the retort. The center tube was raised and is now nine and inches above the peripheral tubes. Valving was also installed to permit changing the ratio of air between the center and peripheral tubes. These changes have proved effective in developing good temperature profiles across the retort.

A center tube was installed for the introduction of a portion of the recycle gas into the center of the bed above the table feeder. This tube was removed shortly after unit startup due to operational problems and channeling of shale flow.

The surge drum has proved effective in settling a portion of the fines ahead of the product recovery system. Shale mass rates have not exceeded 500 lbs/(hr)(ft^2), however, and high fines carryover only becomes a major problem at higher throughputs. No measure has been made of the fines collected in this drum to date.

The electrostatic precipitator has been put into service. As in the case the Retort No. 2 its effectiveness with power both on and off is discussed by the Retort Section.

The air cooled condenser was tested and preliminary indications are that it may be as effective as the electrostatic precipitator. Recycle gas to the retort is visually mist free in contrast to earlier runs.

The unit was down down January 11 for minor repairs and to await the building of an inventory of dry shale sufficient for at least five days operation. This was based on the decision to suspend further test work until the needed dry shale was assured. The shale drying system was installed Tuesday evening January 12. By noon time of January 13, the drying system was deemed successful and the unit was fired on the swing-shift using the available two day supply of dry shale. Shale was loaded into the 60 ton bins from the drying area January 15. Drying of the shale was accomplished using the heat of compression of a 40 HP blower, barn heaters, and shale circulation.

An inspection of the retort during the shutdown revealed the presence of a warp in the south and east wall of the retort above the air-gas distributor. This warp is not of sufficient magnitude to suspend operations for repairs. A new section will be ordered and installed at a convenient time. High temperature skin thermocouples have been installed and skin temperatures are now being recorded. The air-gas center distributor tube was replaced as it has bent to approximately 40° from the horizontal. A gusset support plate was installed to hold the center tube.

Problems are still being encountered with the Richardson weigh system. Complete new components are on order to up-date this equipment and assure its reliability.

Electrical drawings for Retorts No. 1 and No. 2 are being made using the services of Mr. W. B. Magnuson, electrical engineer from the Engineering Group of Socony Mobil Oil Company's Paulsboro Laboratory. This work was completed January 4 to 15.

Work is also progressing in covering the control mechanisms for the shale feed systems under the 60 ton bins. This work will be completed by January 22.

III. RETORTING SECTION (J. E. Lawson)

A. Demonstration Run (I. A. Jefcoat)

A demonstration run was proposed and tried on Retort No. 1. The conditions used were similar to Run 433 which gave a yield of 91% FA. These conditions were:

Raw Shale	500 lbs/(hr)(ft^2)
Particle Size	1/4 - 3/4 inch (APD 0.28
Air	5,000 SCF/T
Recycle	16,000 SCF/T

In addition to producing oil at a high yield, the object of this run was to obtain replicated material balance periods in order to demonstrate improved component balances, and determine the precision with which retorting variables could be controlled. During the run a comparison of the electrostatic precipitator with the condenser-demister for oil mist recovery was to be made.

Discussion of Results

Analysis of the data obtained has not been completed. A summary of the yields and balances are presented below.

Run No.	Yield % FA	Material Balance			
		Overall	Carbon	Hydrogen	Oxygen
454	94.5	98.5	101.2	94.8	93.4
A	74.1	101.3	99.6	118.1	110.0
B	76.0	101.9	97.8	107.2	105.2
C	80.5	102.6	104.0	130.0	72.5
D	79.3	98.1	98.0	96.1	86.5
E	78.8	100.6	106.2	105.3	97.3
F	83.0	100.5	107.6	99.1	95.6
G	75.8	99.5	103.1	99.7	92.8
H	86.2	100.3	113.8	104.8	90.7

The above data are tentative. Runs A through D were calculated without a correction for the water on the raw shale. There is an uncertainty in the analytical determinations of carbon in Runs E through H. These discrepancies are in the process of being resolved.

Comparing results for the two sets indicate an improvement in the overall, hydrogen and oxygen material balances by including the water on the raw shale. The carbon balance for the first set is an improvement over previous runs. This is attributed to the direct measurement of the total carbon in all streams by the Analytical Section. The deviations in the carbon balance in the second set is believed to have been caused by a leak in the apparatus used for carbon determinations.

The liquid product recovered by the electrostatic precipitator was the same with the high voltage on or off. The condenser-demister recovered the same amount of product as the electrostatic precipitator.

Operational Problems

The first week's operation was hampered by a two inch line that was placed in the bottom of the retort to direct part of the recycle gas into the center of the retort. The line was installed about ten inches above the table feeder and was causing the shale to be drawn off faster on one side of the retort

than the other. Run 454 was finally accomplished by directing the majority of the inlet air into the faster moving shale area. This line was removed.

The supply of dry shale was consumed during the first week's operation. Runs A through H were made with muddy raw shale. The water retained on the shale that was fed into the top of the retort was about three weight percent. Efforts were made to adjust the input air to compensate for the water input. This operation was near the flooding point for this APD of 0.28. This hampered the attempts to make an air compensation for the water input. Some improvement was being made as evidenced by the higher yield for Run H.

Carbonate decomposition in excess of 35%, clinkers in the spent shale, and a spent shale Fischer Assay greater than 2.0 Gal/T indicate that the air distribution was not satisfactory.

The retort is being repaired and cleaned at the present time. A sufficient quantity of dry raw shale is being prepared. At the completion of above, an attempt will be made to duplicate Run 454 with a five day period of operation.

B. Retort No. 2 Operations (C. W. Tyson and K. I. Jagel)

As described in the previous Monthly Progress Memorandum, Retort No. 2 was operated in two 9-day test periods to test its capabilities as a research tool and to reproduce runs made by the Bureau of Mines to provide a comparison base point for future research.

Since the last memorandum the second period of operation has been completed and partially worked up. The results from both periods are summarized in Table I. During this latter period three attempts were made to duplicate the yields obtained by the Bureau of Mines. None of these attempts were entirely successful. One additional run at 500 lbs/(hr)(ft^2) was made to probe the improved capabilities of the top disengaging section, modified in the manner described previously. Unfortunately, the improved performance of the disengaging section was more than offset by increased dirtiness of the raw shale feed, the dirt being tightly bound to the shale particles by water and ice. This problem in raw shale quality is presently being worked on with very high priority and will be lessened shortly.

In addition to the improved disengaging section, the electrostatic precipitator was added as the final part of the low pressure recovery system during the second series of experiments in hopes of improving the total product recovery. The electrostatic precipitator recovered almost 50% of the liquid product during this period with or without electrical power as shown in the table below.

Recovery Distribution

n		
rostatic ipitator	H.P. Section	% Oil Lost
in Use	---	---
in Use	---	---
in Use	---	---
in Use	---	---
in Use	---	---
in Use	12.9	4.8
in Use	2.9	6.2
Operation	---	---
Operation	---	---
Operation	---	---
Operation	---	
48.8	1.9	4.3
46.0	0.5	3.9

TABLE I

REPORT NO. 2 - SHAKEDOWN OPERATION AND DEMONSTRATION RUNS (PRELIMINARY RESULTS)

Run No.	Raw Shale Rate (lb/(hr)(ft²)	Total Recycle Rate (SCF/T)	Air Rate (SCF/T)	Dilution Rate (SCF/SCF Air)	Brine[4] Addition (lb/T)	Actual Recovery	Recovery[5]	Yield Corrected For Recovery System Loss	Ash in Oil % Wt	Spent Shale Gal/Ton	Mist P % Oil Recovered I L.P. Section	Mist P % Oil Recovered I Electr Preci
PH43	292[1]	14,500	4,460	0.23	0.0	86.6	93.6	---	0.02	0.35	--	Not
PH44	271	15,900	5,200	0.24	18.1	78.6	76.8	--	0.03	0.35	--	Not
PH45A	271	16,700[2]	5,120	0.24	16.0	78.2	84.3	---	0.02	0.30	---	Not
PH45B	277	16,700[2]	5,010	0.25	14.9	76.4	78.5	---	0.03	0.80	---	Not
PH47	332	15,300	5,840	0.00	19.5	69.9	73.6	---	0.28	0.45	---	Not
PH48	444	11,100	5,391	0.00	25.5	58.3	64.4	67.7	0.14	1.55	82.3	Not
PH49	268	17,100	4,880	0.24	0.0	75.4	83.5	88.5	0.10	0.00	90.9	Not
PH50A	294	16,200	4,510	0.26	17.9	81.0	77.9	---	0.33	0.50	---	In C
PH50B	292	16,300	4,500	0.26	3.6	85.2	87.5	---	0.91	0.35	---	In C
PH51A	298	16,400	4,690	0.32	12.5	64.5	63.3	---	0.33	0.70	---	In C
PH51B	306	14,200	4,570	0.31	17.4	52.2	50.6	---	0.75	0.45	---	In C
PH52	472	15,900	6,170	0.00	17.0	66.3	66.7	69.7	2.18	0.90	45.0	
PH53	288	14,600	5,080	0.23	24.1	63.0	60.0	62.5	0.29	0.50	49.6	

(1) Raw shale size range 1/4 - 1 inch mesh. For all other runs raw shale size range was 1/4 - 3 inch mesh.
(2) Corrected for gas leak out at bottom Star Feeder.
(3) Corrected for air leak in at Electrostatic Precipitator or at top of Retort.
(4) 10% NaCl
(5) 100% ash balance basis assuming that spent shale rate measurement is correct and adjusting raw shale rate.

Raw Shale Rate lb/hr	Elapsed Time, hrs.	Product Rate in ESP lb/hr	ESP Power
4,900	4	149	OFF
4,500	4	142	ON

The reason for its effectiveness as a mist recovery unit appears to have come primarily from the long lines needed to tie it to Retort No. 2, the pressure drop across the unit and its associated piping (approximately 50 inches of H_2O vacuum) and the circuituous path through the electrostatic precipitator holding volume.

One additional problem, suspected during the first operational period and confirmed during subsequent operations, was the significant shale size segregation occurring in the 60 ton bins used to supply Retorts No. 1 and No. 2. This problem was considerably aggrevated by the wide particle size range, + 1/4" - 3", used during most of the operations of Retort No. 2. The cause of this problem is the initial loss of small sized particles from the active volume of the storage bin to the dead areas below the angle of repose of the draw off port. The larger particles remaining created some bridging problems in the feed system and the unit above and beyond those expected from thoroughly-mixed + 1/4" - 3" shale. As the bin was drawn down, the larger sized particles were depleted from the bin leaving behind the smaller particles. As the bin finially emptied, the effluent shale became richer and richer in the smaller particles and dust. The latter created some problems in the mist recovery system. Several solutions have been suggested for this problem. However, the program for the next few months deals strictly with narrow particle ranges which are not sensitive to this defect in the equipment. Therefore the solution of this problem will be held in abeyance until the need arises. As time permits, limited studies will be made in mechanical models to assure the final design is adequate to eliminate this source of difficulty.

As can be inferred from the data presented in Table 1 the process problem of greatest concern was the low oil recoveries obtained. From the four mist recovery determinations performed by K. I. Jagel on Runs B-448, B-449, B-452 and B-453, it is apparent that lack of collection efficiency was not the problem as was previously suspected. Furthermore the average of the carbon material balances available at this writing is 100.3% with a range of -6.3% to +9.3% indicating that all of the carbon entering the system is being accounted for (Table 2). Preliminary analysis indicates that greater than desirable amounts of light hydrocarbons were being produced in the vent gas, presumably as a result of cracking. It is hoped that some idea of the cause of cracking will come from the completion of the detailed data workup presently in progress.

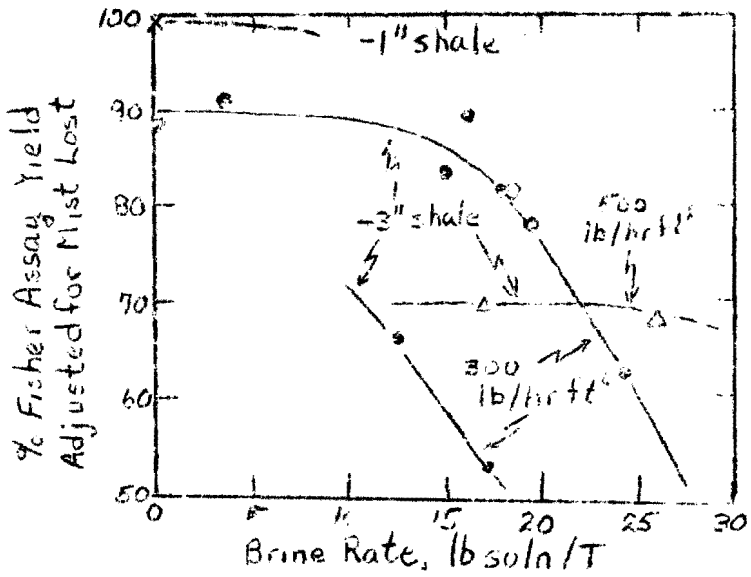
TABLE II

RETORT NO. 2 - SHAKEDOWN MATERIAL AND COMPONENT BALANCES

Run No.	Fischer Assay Yield	Material Balances			
		Overall	Carbon	Hydrogen	Oxygen
B-443	93.7	98.7	103.5	109.9	87.4
B-444	76.8	100.9	103.2	104.3	100.5
B-445A	84.3	100.4	108.1	121.7	90.1
B-445B	78.5	98.2	94.4	100.9	94.3
B-447	73.6	100.4	93.3	106.8	96.9
B-448	64.4	96.1	93.8	94.7	71.9
B-449	83.5	102.6	109.3	130.5	101.7
B-450A	78.0	96.4	91.6	107.8	73.6
B-450B	87.5	103.6	111.2	125.0	104.1
B-451A	63.4	100.6	103.0	118.1	100.3
B-451B	50.6	100.6	95.9	100.1	100.6
B-452	66.7	100.7	105.1	94.7	95.4
B-453	59.8	98.3	98.0	82.2	86.2

1. Ash balance closed, air leak into unit corrected for.

One possible clue is in the apparent deleterious effect on oil yield of brine addition at the 300 lb/(hr)(ft^2) raw shale rate. This is shown in the following empirical plot.



This plot presents several interpretive problems. If the mist recovery system is as efficient as the data presented previously indicate, then brine must be creating a top-of-the-bed refluxing problem. However, consistant gravities around 19 °API were obtained for the liquid products, a level which is usually indicative of little or no refluxing. Furthermore, there is no readily apparent reason for two - 3" shale, 300 lb/(hr)(ft^2) lines. Finally, why does the effect of brine diminish as the raw shale rate increases? The answers to these questions will certainly affect any explanation to the low oil yields from Retort No. 2.

Future Work

As outlined above, the immediate effort will be to continue the completion of the detailed data work up of this series of runs followed by an attempt to understand the events occurring within the retort and within the oil recovery system. Emphasis will be placed on the events occurring in the combustion zone, the top of the retort and in the mist recovery systems since these are the most probable areas of oil yield loss.

C. Mist Formation and Recovery - Size of Oil Mist Droplets (K. I. Jagel)

Two measurements of oil mist particle size distribution have been made using the cascade jet impactor- (The measurements are in addition to the routine measurements of oil mist concentration bypassing the recovery system made with the filter

Figure 1

% Wt Finer than "d"

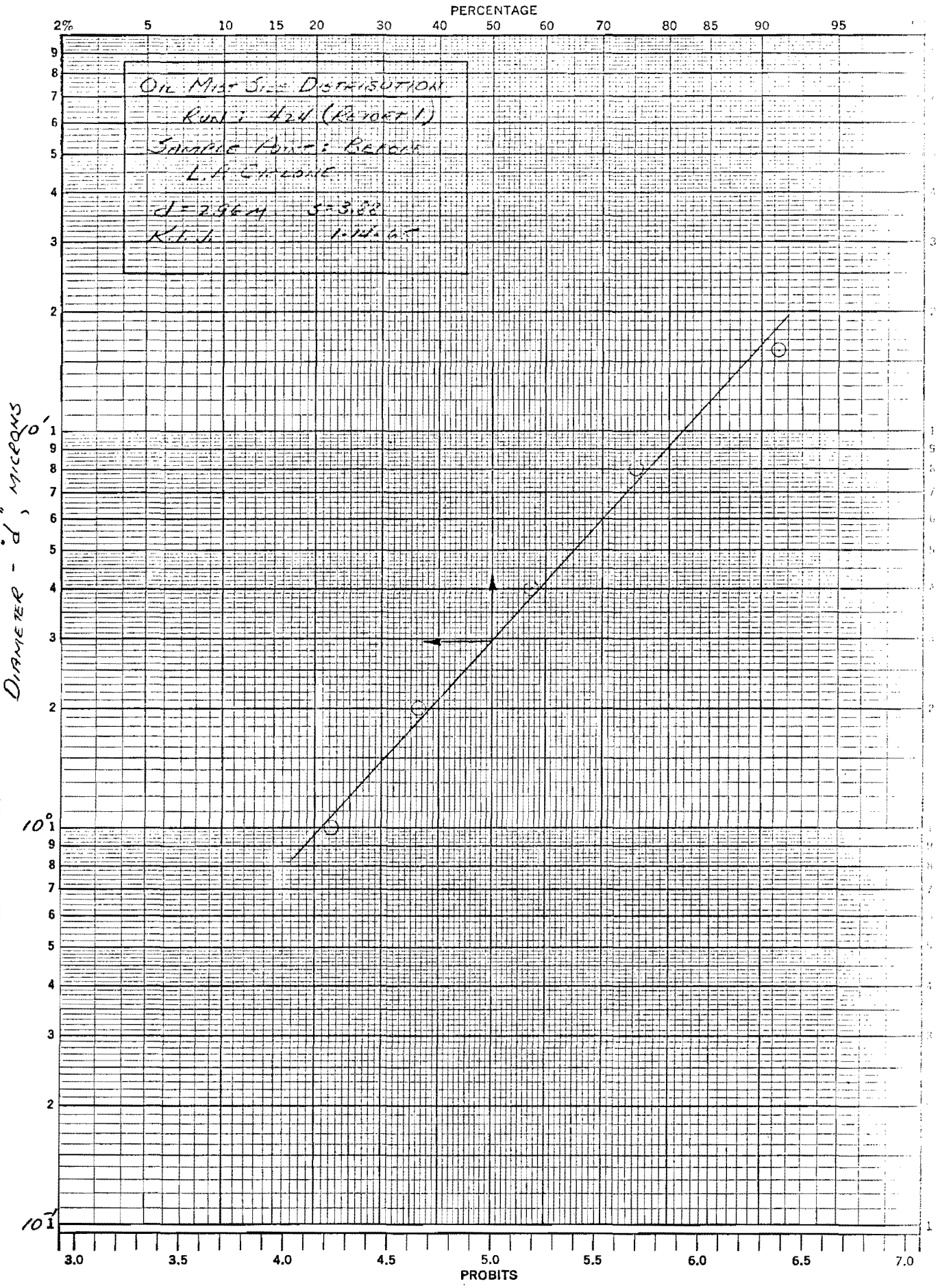
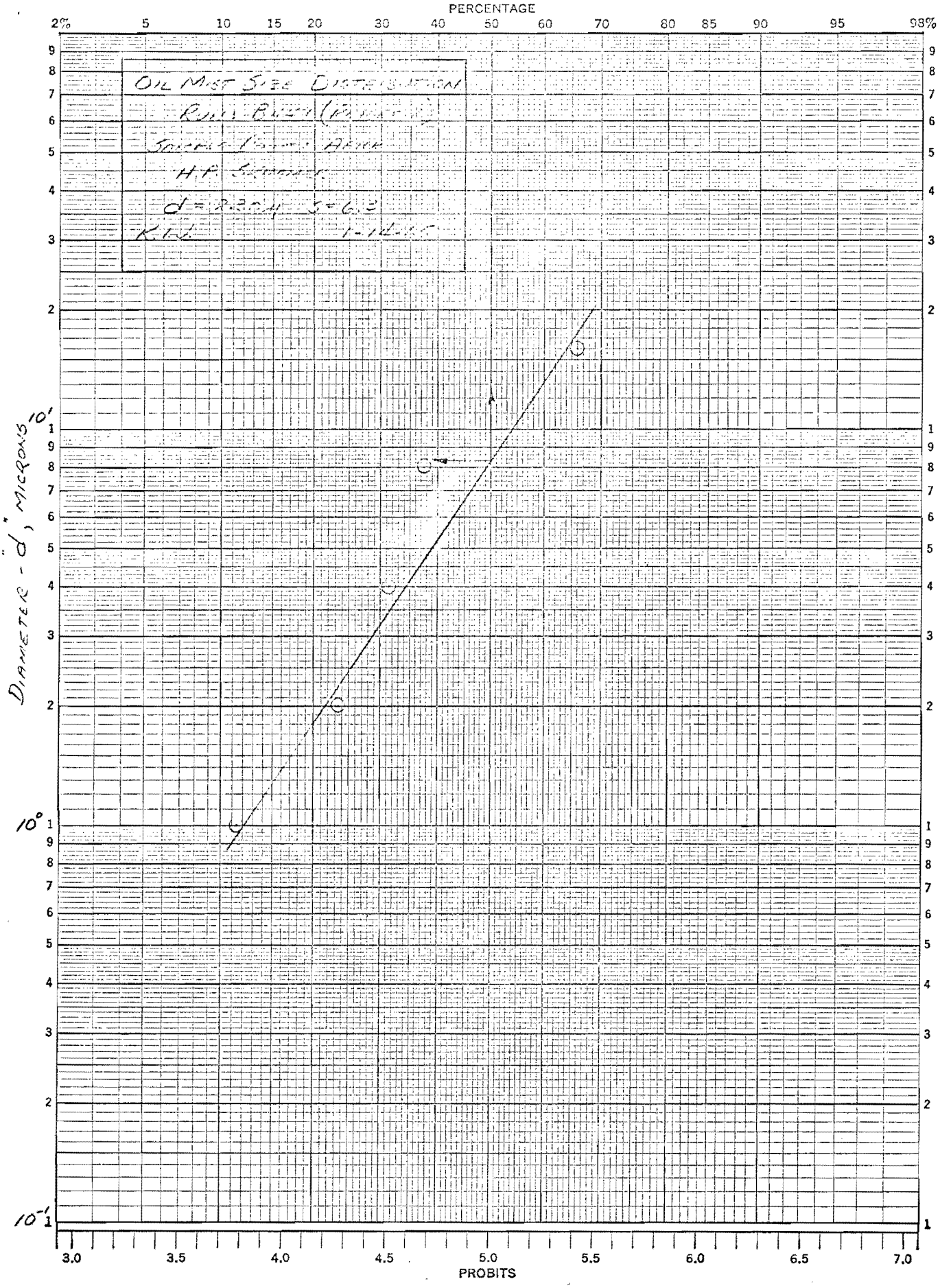


Figure 2

% Wt Finer than "d"



stage of this apparatus.) As has been anticipated, these size distributions are linear when the diameter is plotted as a function of the weight percent finer than that diameter on a logarithmic - probability grid. They are shown as Figures 1 and 2. It is particularly convenient to use this type of plot for summarizing size distribution data since the 50% point is the geometric mean diameter. The width of the distribution, expressed as its standard deviation may be calculated from the ratio of the diameters corresponding to the 84.13% and the 50% points.

The data for Figure 1 were obtained during Run 424 on Retort No. 1. The sample point was the inlet to the low pressure cyclone. This is immediately downstream from the point at which the mist recovery recycle stream and the retort outlet stream join. Brine addition was used in this run. The geometric mean diameter is 2.96 microns with a standard deviation of 3.9.

The second size distribution was obtained during Run B-449 on Retort No. 2. In this case, the sample was taken at the outlet from the high pressure skimmer. No brine was used in this run. The geometric mean diameter is 8.30 microns with a standard deviation of 6.3. The apparently larger size is probably due to the fact that no brine was added with the raw shale and/or that the recycle compressor had an agglomerating effect on the oil mist leaving the low pressure section of the mist recovery train.

These measurements should be considered approximate because of experimental problems in making the measurements. Because of apparent re-entrainment from the various impactor stages it was necessary to assume that no particles smaller than 0.5μ were found in the oil mist. More definitive measurements will be made using an improved experimental technique.

D. Process Variable Study (K. I. Jagel)

The planning for the process variable study which was described in broad outline last month has now been completed. To review briefly, it is planned to optimize the process at particular combinations of shale mass velocity and particle size which will insure reasonable flexibility in evaluating the economic potential of this process. The combinations of shale mass velocity and particle size, the order in which the studies will be carried out and the approximate completion dates for the various studies are presented in Table III.

Because of the sequential nature of the experimental design, it is difficult to detail the runs to be carried out beyond the first nine, nevertheless, a reasonably detailed description of Study I which is to be carried out with 3/4 - 1 1/2 inch shale at 700 lb/(hr)(ft^2) is presented in Table IV.

The variables which will be screened in Phase I of Study I are nucleation, air rate, bed height, air temperature at the retort

TABLE III

Study	Raw Shale Size (inches	Raw Shale Rate lb/(hr)(ft^2)	Completion date Most Likely	95% Certain (Assuming Start on 1-4-65)	Tons of Mine Run Raw Shale Needed and Not on Hand[1] (50% 1 1/2-3" Raw Shale Crushed Mine Run)
I	3/4 - 1 1/2	700	1-30-65	1-30-65	--
II	3/4 - 1 1/2	Maximum Operable Rate	2-14-65	2-18-65	--
III	1/4 - 3/4	500	3-1-65	3-15-65	--
IV[2]	1 1/2 - 3	700	3-27-65	4-14-65	2600
V[2]	1 1/2 - 3	Maximum Operable Rate	4-11-65	5-5-65	5400

[1] It is estimated that there are 2400 tons of 26-28 gal/ton raw shale on hand.

[2] Tentative

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1-14-65

TABLE IV
PROCESS VARIABLE STUDY

Study I Raw Shale Rate: 700 lb/sq ft-hr Raw Shale Size Range: 3/4 - 1 1/2" Retort: No. 1

Phase I - Initial Screening Study

Variables:		Nucleation Air Rate (Gal/Ton RS of 10% NaCl)	Air Rate (SCF/Ton RS)	Bed Height Above Air Distributor (Ft)	Air Inlet Temperature (°F)	Total Recycle Gas Rate (SCF/Ton RS)	Dilution Gas Rate (SCF/SCF Air)	Remarks
Levels of Variables:								
	+1	5	5500	7	1200	18000	0.8	
	0	3	4750	6	900	16000	0.4	
	-1	1	4000	5	600	14000	0.0	
Runs:	1	0	0	0	0	0	0	Estimated Optimum Before Study
	2	-1	-1	-1	-1	-1	-1	Most Economical in Utilities and Investment
	3	+1	-1	+1	-1	+1	-1	
	4	-1	+1	-1	+1	+1	-1	
	5	+1	+1	+1	+1	-1	-1	
	6	+1	-1	-1	+1	-1	+1	
	7	-1	-1	+1	+1	+1	+1	
	8	+1	+1	-1	-1	+1	+1	
	9	-1	+1	+1	-1	-1	+1	

Phase II - Line of Steepest Ascent Study

Two runs will be made along line of Steepest Ascent calculated by Box Method from Phase I results

Phase III - Description of Experimental Region Near Optimum

Variables:		A'	B'	C'		A'	B'	C'
		Part A					Part B	
Runs:	1	0	0	0	Runs: 10	-1.215	0	0
	2	-1	-1	-1	11	-1.215	0	0
	3	+1	-1	-1	12	0	-1.215	0
	4	-1	+1	-1	13	0	+1.215	0
	5	+1	+1	-1	14	0	0	-1.215
	6	-1	-1	+1	15	0	0	+1.215
	7	+1	-1	+1				
	8	-1	+1	+1				
	9	+1	+1	+1				

(The three variables chosen for this Phase are the three most significant variables from Phase I)

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1-14-65

inlet, recycle gas rate and dilution gas rate. This screening phase is a one-eighth replicate of a 2^6 factorial experiment plus a single run at which all of the variables are held at their base level. At the end of Phase I the following alternatives should be considered:

1. If Run 1 yield is equal to the mean of Run 2 to 9 yields, the Box Method of Steepest Ascent to determine conditions for two Phase II runs which should increase yield should be used.

2. If Run 1 yield is not equal to the mean of Run 2 to 9 yields, and
   a. no yield is greater than 85%, the Box Method of Steepest Ascent to determine conditions for two Phase II runs which should increase yield should be used. If these runs do not increase yield the experimental design is in the region of the optimum. The first nine runs are then to be considered Phase III A. The alternatives to be considered at the end of Phase III A should be evaluated at this point.
   b. any yield is greater than 85%, it should be considered that the Phase I runs were carried out in the region of the optimum. The first nine runs are then to be considered Phase III A. The alternatives to be considered at the end of Phase III A should then be evaluated.

Phase III of Study I, which is intended to describe the effect of the process variables in the region of the optimum, is to be carried out with the three most significant variables from Phase I. This phase is an orthogonal composite design with three independent variables. When Phase III A is completed the following alternatives should be considered:

1. If Run 1 yield is equal to the mean of Run 2 to 9 yields, the region of the optimum is relatively flat and no further experimentation is required to describe the region.

2. If Run 1 yield is not equal to the mean of Run 2 to 9 yields, the region of the optimum is highly curved and Phase III B must be carried out to adequately describe the region.

E. Mechanical Models (T. C. Lyons and L. J. Skowronek)

1. Shale Flow Studies

The basic flow characteristics were determined for a nominal 1 1/2 - 3 inch shale size. This information is summarized in Table V along with data obtained previously for the 1/4 - 1 inch and 1/4 - 3 inch material. In all cases, larger openings were required to insure continuous flow than were necessary with the full size range (1/4 - 3 inch).

TABLE V

BASIC FLOW CHARACTERISTICS OF OIL SHALE

Nominal Size Range, Inches	1/4 - 1	1/4 - 3	1 1/2 - 3
Average Particle Diameter, Inches	0.55	0.57	1.1
Bulk Density, lb/cu ft			
Loose Poured	65.2	73.8	70.7
Packed	71.9	80.7	77.0
Minimum Diameter of Circular Opening to Insure Continuous Flow, Inches	.5	12	14
Minimum Width of Rectangular Slot to Insure Continuous Flow, Inches[1]	3	10	14
Angle of Flow (With Horizontal) °	42 - 43	39	--
Angle of Repose (With Horizontal) °	40 - 41	39	--
Angle of Internal Friction (Coning Angle With Horizontal) °	70	60	--

[1] Slot was 14 3/4 inches long in all cases

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Initially, the 1 1/2 - 3 inch size range was studied to combat a segregation problem in the shale inlet system of Retort No. 2 (December Memorandum). However, since that time it was decided to include this size as a standard cut in the proposed process variable study. Therefore, the entire spectra of flow characteristics were determined. Motion pictures have been made of the flow patterns and the angular properties will be determined from the pictures.

Although the shale was initially screened at the crushing plant to reject material smaller than 1 1/2 inch, it contained about 15% of dust, chips, and fines during the flow studies. This was the result of normal attrition in the shale handling system at the crushing plant and in the flow studies themselves. This accounts for the low average particle diameter (1.1 inch) and demonstrates the tendency for shale particles to dust and chip during handling.

In order to evaluate the 1 1/2 - 3 inch size, it was necessary to increase the outlet of the shale flow model from 12 to 14 inch I.D. As time permits, the flow capacity curves for the 1/4 - 1 inch and 1/4 - 3 inch cuts will be extended to include the larger pipe size.

2. Air Distributor Model

The air distributor model - for investigating gas mixing and distribution in a shale bed - has been constructed and the shakedown studies are underway. Up to this time it has not been possible to get sufficient smoke concentration in the air streams to trace the flow pattern adequately. Attempts are being made to overcome this problem by purchasing larger smoke candles in addition to making mechanical changes in the smoke distribution system.

IV. ANALYTICAL LABORATORY SECTION (B. L. Beck and D. Liederman)

A. Gas Analysis

Two 44 liter gas holders have been installed in the laboratory and connected (via heated copper tubing) to Retorts No. 1 and No. 2. The present gas sampling procedure is to continuously sample the recycle gas into a gas holder; the sampling rate is adjusted so that the holder is almost full after a twelve hour test period.

In addition to the gas chromatographic analysis of the gas, we are also determining total carbon and hydrogen by combustion and absorption of CO_2 and H_2O. Early results indicate that this procedure is significantly helping to solve the carbon balance problem.

B. Quality Control Program

The second phase of the quality control program, that of continually and systematically running tests on control samples began January 4th. A schedule has been prepared for each technician to run different quality control tests each week. The control testing amounts to approximately 5% of the sample load. The frequency with which tests are checked varies from a maximum of three times a week to a minimum of once every two weeks, depending upon how often the test is required in sample testing.

C. Carbon-Hydrogen Analysis of Carbonates

As a further check on the ability of the carbon-hydrogen procedure to completely decompose carbonates in shale samples, two standards were run. One was reagent grade basic magnesium carbonate and the other was NBS argillaceous limestone. The results given below show that we are completely decomposing the carbonates. This supports the C-H comparisons with the Humble Service Laboratories and the USBM at Laramie discussed in the last memorandum.

Material

	Carbon Wt. %		Hydrogen Wt. %	
	Present	Found	Present	Found
Basic Magnesium Carbonate	9.9	10.0	2.20	2.30
Argillaceous Limestone	9.76	9.81, 9.64	--	--

D. Miscellaneous

Beginning January 4 the laboratory started on a seven day operating schedule covering the day and swing shifts.

All five of the laboratory technicians are now trained to run all of the tests in operation in the laboratory.

Tentative procedures have been written for all laboratory tests.

Last June an estimated 1964 budget of $14,000 for laboratory equipment, supplies, and chemicals was prepared. As of December 31, 1964 the total of the laboratory's purchase orders was approximately $13,600.

V. ENGINEERING AND ECONOMIC ANALYSIS (P. W. Snyder and J. E. Burchfield)

1. Retort No. 2 Initial Operating Performance as Measured by Material Balances

The material balances for our first 13 runs on Retort No. 2 have been analyzed. They are summarized and compared with

Retort No. 1 and the Bureau of Mines performance in Table VI. A great deal of difficulty was experienced with the raw shale feeding system because of fines and wet shale; and the more precise gas composite sampling system and direct carbon analyses were available only for the last three or four runs. Therefore, the performance was as good as could be expected, but not what we hope to achieve on this unit.

The revisions made to the pilot retorts in order to improve the reliability of the material balances and oil yield are listed in Table VII.

Retort No. 1 will be run at a single set of operating conditions for a one week period to confirm the reproducibility of yields, operating conditions and to obtain a sufficient amount of data to establish the overall precision of our revised sampling and analytical procedures.

2. Effect of Sampling Precision on the Reliability of Reported Oil Yield

A series of material balances were calculated for Run 432 in which the major input data were varied over the estimated range of precision for the initial sampling procedure as well as over the expected range for the revised procedure. The results of this study are shown in Table VIII.

The observed ± 15% for the 95% confidence in the carbon balances for the past runs appears to be principally due to poor precision of the raw shale rate, carbon on raw and spent shale, and carbon in the gas. The revised procedure which is aimed at improving these precisions should reduce the deviation in carbon balance to ± 6% in Fischer Assay yield; the precision of the oil collected in the tanks is ± 3% of the Fischer Assay based on ± 1% precision of the raw shale rate and ± 0.5 gal/ton precision of the Fischer Assay.

Therefore, actual oil yield should be never less than 3% of Fischer Assay from the reported value or any higher than 6% Fischer Assay from the reported value. In other words the reliability of reported oil yield is + 6%, - 3% of Fischer Assay for 95% confidence. (The + 6% is due to the possibility of oil loss through the recovery system and the resulting reliance on carbon balance for accountability.)

3. Math Model Simulation of Current Runs

The following pilot retort runs from Retort 1 have been simulated with the math model.

TABLE VI

MATERIAL BALANCE PRECISION FROM PILOT RETORTS

Retort Run Number	Current Performance				Bureau of Mines Performance	
	No. 1			No. 2	No. 1	No. 2
	222 to 406	408 to 425	426 to 442	443 to 451[1]	Picked at Random	14 to 21
Number of Balances:	18	19	14	5	18	22
Ash Balance						
Mean	97.4	96.2	95.9	97.8	99.3	--
95% Conf. Limits	±13	±13	± 8	±12	± 8	--
Overall Balance						
Mean	99.8	99.6	100.5	100.1	101.4	101.4
95% Conf. Limits	± 5	± 5	± 3	± 3	± 3	± 9
Carbon Balance						
Mean	96.0	105.5	101.2	102.4	93.6	94.6
95% Conf. Limits	±13	±16	±15	±16	±16	±20
Oxygen Balance						
Mean	--	93.8	101.3	97.1	--	--
95% Conf. Limits	--	±20	± 8	±13	--	--
Hydrogen Balance						
Mean	--	107.5	117.8	110.4	--	--
95% Conf. Limits	--	±29	±22	±23	--	--

[1] Material Balances completed on only 5 out of 13 test periods.

TABLE VII

REVISIONS MADE TO RETORT NO. 1 AND 2 TO IMPROVE MATERIAL BALANCE ACCURACY AND PRECISION

A. Raw Shale System

1. Installed integrator on Syntron rate controller.
2. Began reading mechanical counter on batch dumps from Richardson Batch Weigher.
3. Weather protection and shale drier at 60 ton raw shale bins are being installed.
4. Dust screening system at Utah Feeder is being installed

B. Spent Shale System

1. Installed automatic spent shale sampling system to collect composite sample.

C. Gas Sampling

1. Installed gas sampling line with condenser-filter system. This system is being further developed to obtain quantative measurements of water and oil mist contents of the vent gas.
2. Installed gas holder to permit collection of composite gas sample.
3. Established analytical test to measure carbon and hydrogen in the gas directly by combustion.
4. Established the jet impactor test as routine for oil mist analyses of vent gas.
5. An oxygen analyzer and a continuous chromatograph monitor are being considered to improve the control and definition of when the unit is lined out.
6. A humidity measuring instrument is being purchased to measure directly the vapor content of the vent gas.

D. Oil Recovery System

1. Connected the Electrostatic Precipitator in parallel down-stream to existing recovery train.
2. Installed a settling chamber at offgas outlet from retort ahead of existing recovery train.
3. Installed a gas cooler - oil demister system in parallel to existing recovery train.
4. Installed oil skimmer just upstream of recycle gas blower.
5. Installed water decanter to remove excess water from oil stream.

TABLE III

EFFECT OF SAMPLING PRECISION ON THE RELIABILITY OF REPORTED OIL YIELD

Test	Test Precision: 95% Conf. Limits		Effect on Precision of Material Balance, Wt %: Carbon		Oxygen		Hydrogen	
	Initial Method	Goal for Revised System	Initial Method	Revised System	Initial Method	Revised System	Initial Method	Revised System
A. Raw Shale and Spent Shale Analyses[1]								
1. Rate, %	± 5.0	± 1.0	∓ 5.0	∓ 1.0	∓ 3.7	∓ 0.7	∓ 5.0	∓ 1.
2. Total Carbon, Wt %	± 0.7	± 0.3	∓ 4.0	∓ 1.5	--	--	--	--
3. Mineral CO_2, wt %	± 1.3	± 0.6	--	--	± 4.7	± 2.2	--	--
B. Gas Analyses								
1. Total Carbon, lbs/MSCF	± 2.3	± 0.2	± 4.5	± 0.4	--	--	--	--
2. H_2, lbs/MSCF	± 0.5	± 0.1	--	--	--	--	± 8.7	± 1.7
3. CO_2, vol %	± 4.4	± 2.0	∓ 2.9	--	± 5.8	± 2.6	± 8.5	--
4. H_2O, lbs/MSCF	± 3.0	± 0.5	--	--	± 4.0	± 0.7	± 5.4	± 0.9
Maximum Deviation			±13.5	± 2.9	±18.2	± 6.2	±19.1	± 3.6
Observed Deviation			±15.0		±14		±25	
Unaccounted for Deviation			± 1.5		--		± 6	

(1) Maximum difference between raw and spent shale properties

Run Number	415	420	431	439
Height Above Air Inlet, ft	6	6	5	4
Shale Rate, lbs/(hr)(ft^2)	277	490	630	470
Air Rate, SCF/T	5,500	5,500	4,800	5,300
Total Recycle, SCF/T	17,000	17,000	15,000	14,500
Dilution Gas, SCF/T	2,400	2,450	0	0
Air-Dil. Gas Preheat, °F	1,020	1,030	Amb.	Amb.

In order to obtain solution with the model it was necessary to use 1.0-inch average particle size instead of the measured 0.4 inch. Attempts with the small size brought the shale and gas temperatures so close that the inherent noise in the program made convergence impossible. The calculated pressure drop was corrected for this difference. The results of these simulations are summarized in Tables IX, X, XI, and XII.

The most significant observations are:

1. The model reproduces the overall temperature profile quite well - when the measured peak temperature moves up the bed from the air inlet the model also shows this shift (Runs 415 and 420 vs Run 431).

2. The model does not agree with the measured temperatures one foot above the air inlet. I suspect this probe is reading low, however, a brief calibration test indicated that it should read correctly. It has been replaced and the leads will be checked. This unexpected temperature difference will be examined on future simulations.

3. The calculated mean spent shale temperatures are higher than the measured spent shale temperatures by 60 to 125 °F. The calculated surface temperatures are 30 to 90°F higher than the measured temperature. It appears that the gas temperature is influencing our spent shale temperature measurement.

4. The carbonate decomposition calculated by the model is much lower than the measured decomposition, this is probably due to the larger particle size used in the calculation and to a too high net heat of reaction used for carbonate decomposition. Improvements will be made to the model to bring this difference in line.

The first series of the process variable study is being simulated on the Math Model. The results will be used to assist in guiding the program.

B. Economic Analysis

No economic analyses were made during December.

TABLE IX

UMMARY OF MATH MODEL RESU[illegible]S

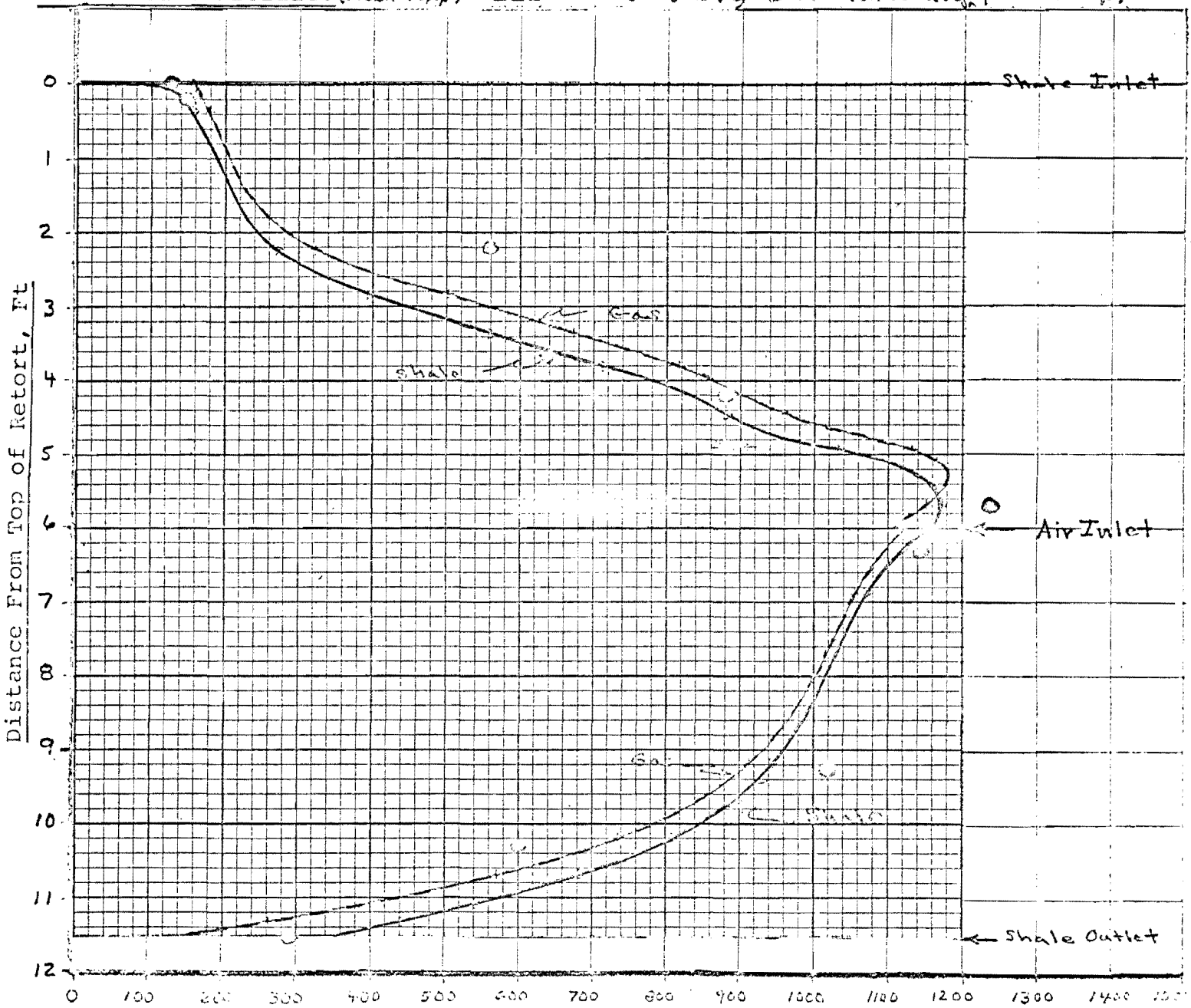
Run No.: 415

Operating Conditions

Raw Shale Rate, lbs/(hr)(ft^2)	- 490	Dilution Gas, SCF/T	- 2400
Raw Shale Size, inches A.P.D.	- 0.4 (1)	Brine Addition, lb/T	- 20
Air Rate, SCF/T	- 5500 (2)	Air-Dil Gas Preheat, °F	- 1020
Total Recycle Gas, SCF/T	- 17,000		

Results		Pilot Retort	Math Model
Shale Oil Yield, vol % Fischer Assay	-	91.3	—
Kerogen Decomposition, %	-	100	100
Carbonate Decomposition, %	-	29	14
Pressure Drop [illegible]," H_2O/ Ft	-	1.6	1.1 (1)
Dry Gas Heating Value, Btu/SCF	-	—	58
Maximum Temperature Across Particle, °F	-	—	63
Length of Combustion Zone, Ft	-	—	1.8
Oxygen Used to Burn Coke, %	-	—	41

Temperature Profile: (Mean Temp) ═══ Math Model; ○ measured avg. probe temp.



(1) Regd 1.0 "APD to obtain solution with math model. AP corrected to 0.4" APD

(2) Air rate corrected for leak.

TABLE X

JMMARY OF MATH MODEL RESU[ ]

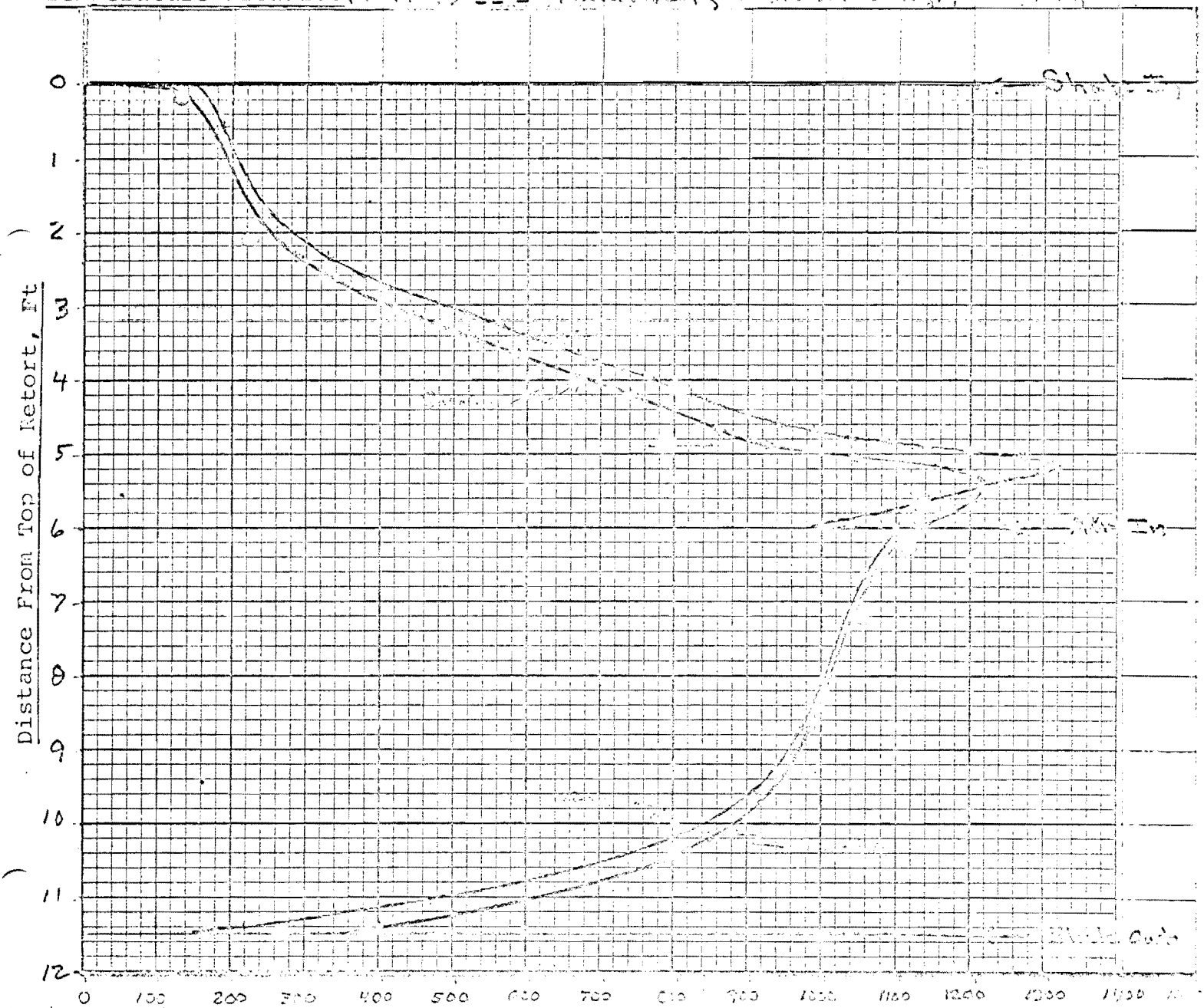
Run No.: 420

Operating Conditions

Raw Shale Rate, lbs/(hr)(ft^2)	277	Dilution Gas, SCF/T	2450
Raw Shale Size, inches A.P.D.	0.45 (1)	Brine Addition, lb/T	0.0
Air Rate, SCF/T	5500 (2)	Air-Dil Gas Preheat, °F	1030
Total Recycle Gas, SCF/T	17000		

Results	Pilot Retort	Math Model
Shale Oil Yield, vol % Fischer Assay	95.8	—
Kerogen Decomposition, %	100	100
Carbonate Decomposition, %	20	22
Pressure Drop, " H_2O/ Ft	0.5	0.3 (1)
Dry Gas Heating Value, Btu/SCF	—	58
Maximum Temperature Across Particle, °F	—	48
Length of Combustion Zone, Ft	—	0.7
Oxygen Used to Burn Coke, %	—	30

Temperature Profile: (Mean Temp) ——— Math Model; ○ measured avg. probe temp



(1) Required 1.0" H$_2$O to obtain calculated recycle rate

(2) Corrected for air leak

TABLE XI

SUMMARY OF MATH MODEL RESULTS

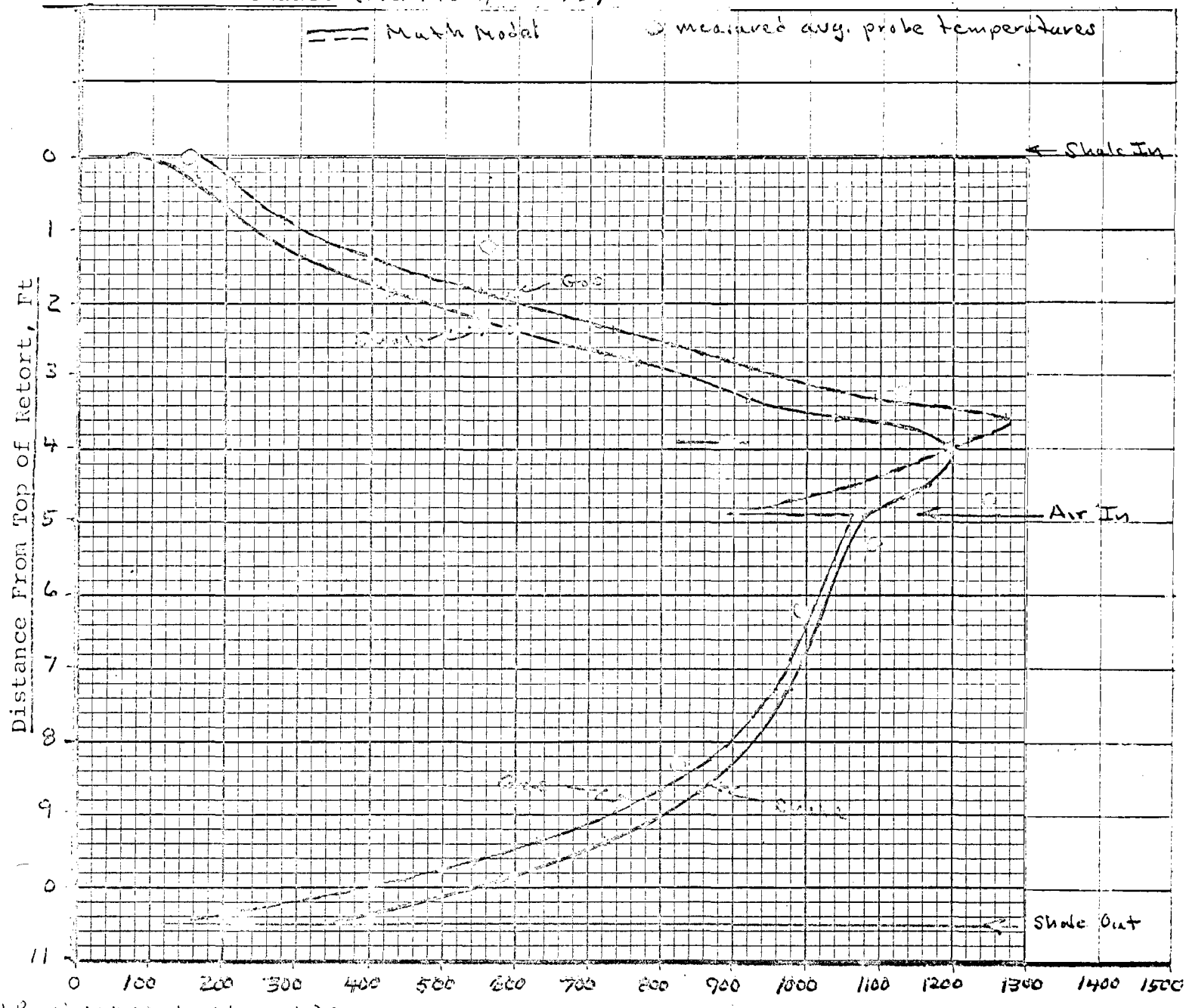
Run No.: 431

Operating Conditions

Raw Shale Rate, lbs/(hr)(ft^2)	630	Dilution Gas, SCF/T	0.0
Raw Shale Size, inches A.P.D.	0.42 (1)	Brine Addition, lb/T	0.0
Air Rate, SCF/T	4800	Air-Dil Gas Preheat, °F	ambient
Total Recycle Gas, SCF/T	15,000		

Results	Pilot Retort	Math Model
Shale Oil Yield, vol % Fischer Assay	81.1	—
Kerogen Decomposition, %	100	100
Carbonate Decomposition, %	24	12
Pressure Drop, " H_2O/ Ft	2.3	1.2 (1)
Dry Gas Heating Value, Btu/SCF	126	69
Maximum Temperature Across Particle, °F	—	69
Length of Combustion Zone, Ft	—	1.9
Oxygen Used to Burn Coke, %	—	48

Temperature Profile: (Mean Temperatures)



(1) Reset 1.0" APD to obtain solution on Model

TABLE XII

UMMARY OF MATH MODEL RESU S

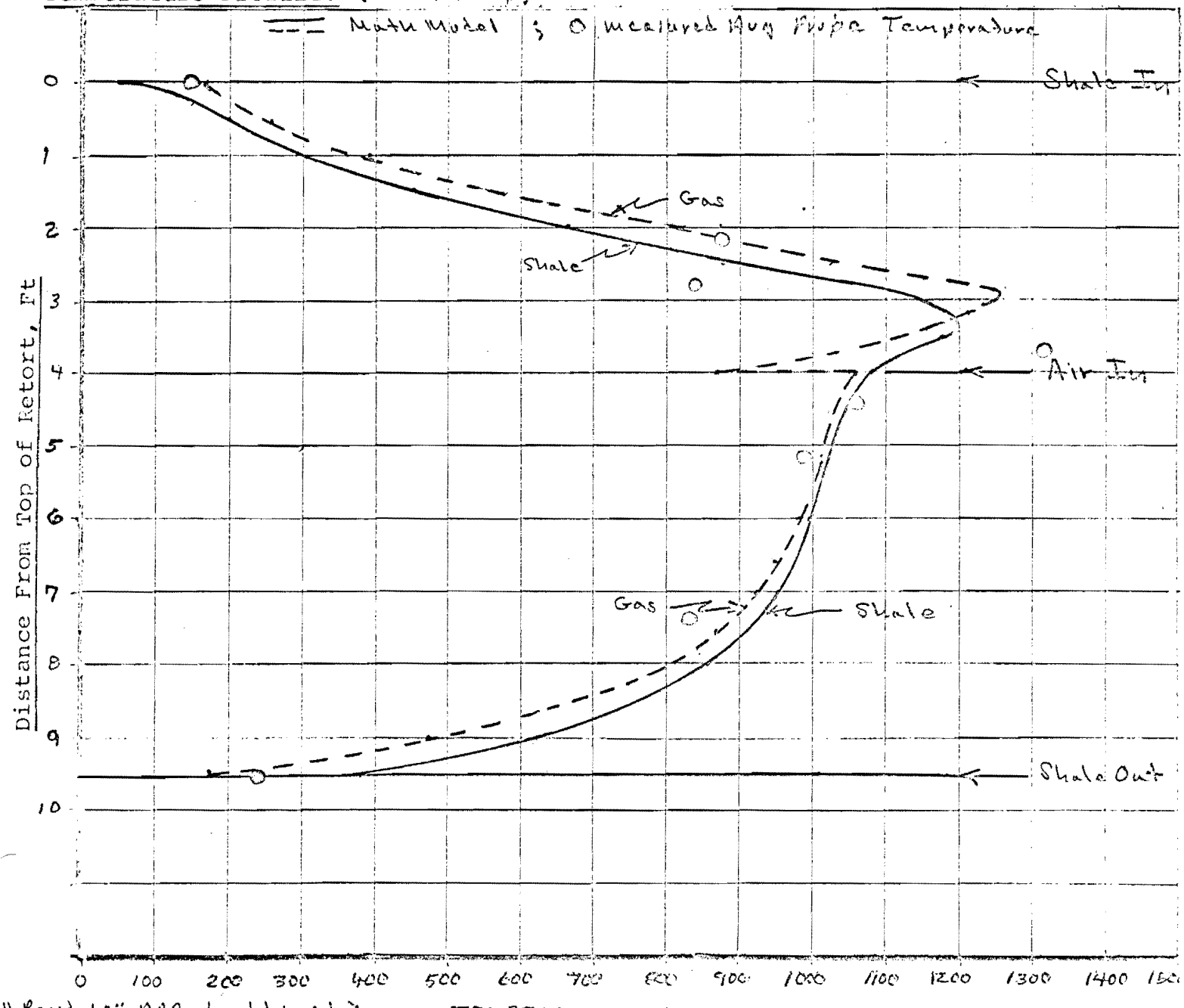
Run No.: 439

Operating Conditions

Raw Shale Rate, lbs/(hr)(ft^2)	470	Dilution Gas, SCF/T	0
Raw Shale Size, inches A.P.D.	0.3901	Brine Addition, lb/T	0
Air Rate, SCF/T	5380	Air-Dil Gas Preheat, °F	ambient
Total Recycle Gas, SCF/T	14500		

Results	Pilot Retort	Math Model
Shale Oil Yield, vol % Fischer Assay	89.9	—
Kerogen Decomposition, %	100	100
Carbonate Decomposition, %	36	14
Pressure Drop Above Air Inlet, " H_2O/ Ft	1.2	0.9 (1)
Dry Gas Heating Value, Btu/SCF	120	80
Maximum Temperature Across Particle, °F	—	64
Length of Combustion Zone, Ft	—	1.6
Oxygen Used to Burn Coke, %	—	50

Temperature Profile: (Mean Temp)



(1) Regid 1.0" APD to obtain solution with math model, DP corrected to 0.39"