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NOTICE

The information contained in this report is regarded as confidential and proprietary. It is provided subject to the provisions regarding confidential, proprietary information contained in the Research Agreement among the Participating Parties.

SOCONY MOBIL OIL COMPANY, INC.

RESEARCH DEPARTMENT

MONTHLY PROGRESS MEMORANDUM
(Covering August 16 to September 15, 1965)

ANVIL POINTS OIL SHALE RESEARCH CENTER

Rifle, Colorado

September 24, 1965

CONTRIBUTORS:

Mining Section
Mechanical Engineering
Retorting Section
Analytical Laboratory Section
Engineering and Economic Analyses

Signed by:

R H Cramer
R. H. Cramer
Program Manager

FRC

PRODUCTION RESEARCH
Return to File Indicated

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FRC		
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JTG		
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GJH		
JEL		
GB		
JR		

September 30, 1965

File 108.04

Mr. J. H. Smith
Process Engineering
Continental Oil Company
Ponca City, Oklahoma

Dear Mr. Smith:

ANVIL POINTS OIL SHALE PROJECT
MONTHLY PROGRESS MEMORANDUM
(Covering August 16 to September 15, 1965)

Each to

Herewith are two copies of the subject report for your distribution.

HEG

Yours truly,

R. H. Cramer
Program Manager

rl
Attachments

cc: W. L. Jensen

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

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MONTHLY PROGRESS MEMORANDUM
(Covering August 16 to September 15, 1965)

I. ADMINISTRATIVE

There have been significant personnel changes during the past month. Humble has rotated people in three positions:

Analytical Laboratory Section

R. Bernheimer replaced B. L. Beck as an Analytical Chemist. (D. Liederman was appointed Supervising Chemist at Anvil Points with Humble's concurrence.)

Retort Section

E. E. Turner and D. P. Cotrupe replaced C. W. Tyson and I. A. Jefcoat as Retort Engineers.

In addition, Phillips has assigned R. L. Clampitt as a full time Technical Observer at Anvil Points. Mr. Clampitt is working with the Retort Group.

Breaking these new people in and acquainting them with Research Center procedures are proceeding rapidly and smoothly, although it does impose some burden on the remaining members of the technical staff.

II. MINING SECTION (G. R. Haworth and J. B. Sellers)

A. Production

The fifth and final round in "Charlie" was drilled and blasted on September 8. Improved fragmentation resulted from more careful attention being paid to blast hole positioning. The yield was 2,350 tons of oil shale, and haulage to the crusher plant has started. All future rounds will be taken from the north end of "Able".

B. Development

Work on the development necessary to open up a full 40 foot high face at the north end of "Able" is complete, with the exception of loading out the last round. The floor in "Able" and Crosscut-9 was 9 to 11 feet above the floor in "Charlie". A 25 foot wide cut has been benched out along Crosscut-9 from "Charlie" to "Able". A 40 foot wide cut has been benched out to the face at the north end of "Able", with a ramp connecting the lower and original floor.

Blast hole patterns were varied during the benching program. Work was started with 4 foot 6 inch spacing and burden, and later increased to 6 foot centers. The last round has been drilled with holes inclined at 30° to the vertical in an attempt to reduce the number and size of large slabs of shale in the blasted rock. Examination of the shale as it is loaded out of the face will indicate whether inclined holes should be investigated at a later stage, during bench blast research work. The tonnage of shale finally to be removed from this 9 to 11 foot bench totals 4,700 tons, averaging 47 gal/ton. This shale is being transferred to Crosscuts 1 and 2 where it is stockpiled for any possible future use.

C. Water Supply System

Work is continuing on rehabilitating the mine water supply system. This system is supplied by reservoirs above the mine on top of the mesa. A pump at the reservoirs feeds a head tank from which piping leads down a borehole into Adit No. 4, and on to Adit No. 1.

The pump house has been repaired and the transmission line to the pump house is under repair. The pipeline between Adits 1 and 4 has been checked out, and the foot path from the ventilation raise to the top of the cliff face has been put back in good condition for pump servicing purposes. There are eight more days of electrical work to be completed before the pump can be connected. The surface piping system can then be checked out and put into operation. Water haulage from the plant by truck will be discontinued.

D. Mine Road Maintenance

The portable crushing and screening plant has been overhauled and is to be put into operation, crushing gravel, on September 20. The road between the main highway and the Anvil Points plant has been resurfaced and therefore gravel haulage to the mine road can be resumed.

E. Mining Research

The sag rods have been delivered from the manufacturer and are being prepared for installation. Holes for the rod installations have been drilled in the roof of "Able" haulageway at all crosscut intersections.

Sag measurements taken from these rods will give an indication of any movement or parting of the bedding planes in the roof of "Able" haulageway.

III. MECHANICAL ENGINEERING (W. S. Bergen)

Retort No. 2 was evaluated using the riser type distributors and the liner. During this period a major shutdown for ten days was required to repair the primary crusher.

Retort No. 1 was revised for the use of oxygen for combustion air to obtain a high Cp gas.

Summary

The addition of the liner appears to have eliminated areas of low density in the bed. Yields of 86 to 87% were found at two operating conditions.

Clinkering has developed on occasions usually following a shale stoppage or loss of combustion.

The primary crusher repairs extended to a ten day period, shutting the retort down for a week. All main bearings of the crusher were rebabbitted and the Pitman Cap cracks rewelded.

Revisions to Retort No. 1 were completed and the unit put into service September 12.

Discussion

1. The riser distributors and the liner were tested at several key conditions producing yields to 87%. During the last runs, the riser ports were fouled and a clinker was formed. The exact time this occurred is not known. However, fouling has occurred when unretorted shale has reached the combustion zone. This condition did occur when lining out for the very low air, high recycle run.

   No warping or bending of the distributor headers or risers have occurred. These risers were connected to the air headers by thread connections just below the combustion zone. After two months of service, they were removed with no galling of the threads. "Silver Goop" manufactured by Crawford Mfg. Co. is used as the anti galling compound. New slotted, low velocity port risers will be installed for operation the week of September 19, 1965.

2. New risers for Retort No. 2 were designed. Air will discharge from the riser slots with a 35 to 50 ft/sec velocity. This contrasts the high velocity port design just removed. The low velocity ports are being tested to ascertain port velocity effects upon dust generation, combustion intensity and yield.

3. The electrostatic precipitator has functioned with no problems. The addition of the second support has proved effective and efficiencies are maintaining the 98.5% levels.

4. The liner has warped badly in the combustion zone. It has been straightened on each major shut down. A gradual coke build-up is occurring behind the liner in the retorting zone. It will require removal and cleaning on the next shutdown. The liner in the retorting zone has proved effective in minimizing shale bridging and eliminating the low density bed.

5. The cyclone liquid recovery has varied from approximately 60 to 85+% depending upon the recycle gas rate. At low gas rates, the cyclone is most inefficient. This contributes to fouling during startup. One solution to the startup problem mentioned in last month's report is to size a cyclone for startup conditions. Other recovery equipment is being investigated which hopefully will be more efficient at wider operating ranges.

6. Mist recovery equipment now contemplated for testing are:
   a. Cyclone (in use)
   b. Multiclone (being fabricated)
   c. Floating bed scrubber (to be rented)
   d. Venturi type scrubber (to be borrowed)
   e. Rotoclone (on site)
   f. Electrostatic Precipitator (in use)
   g. Demister (tested)
   h. Air cooled condenser (tested)

7. During the shutdown for the crusher repairs, one roll of the spent shale roll feeder was found to be loose on its drive shaft. It was repaired. However, the implications of the loose roll on past runs must be judged. The loose roll was not completely friction free from the shaft and would move at random. The chance of uneven shale flow east to west was very probable. Charts of the Gamm-O-Tron level control show the effect of the repair. Variation in roll speed is now minimum.

   A review of the Gamm-O-Tron charts indicates the break may have occurred between Runs B445 and B446, last December. Clinkering problems during past operations may also have been caused by short time slow downs of the east roll.

8. The dilution gas for Retort No. 2 is now taken from the purge condenser effluent before the reheat section. This change was made August 16. The revision was made to provide the cleanest possible gas to the risers thereby minimizing port fouling.

9. Liquid and dust sampling problems are recurring. In order to remove differences in unit operator techniques in taking the liquid sample, a True-Cut proportionate fluid sampler was purchased for D-3 and T-3 tanks. Delivery is expected the week of September 19.

Facilities to sample dust from the Rotoclone exhaust are being provided and will be operative the week of September 19.

10. Present plans are to test horizontal pipe distributors in Retort No. 2 after the conclusion of the low velocity riser tests. The results of the tests with the lower slot velocity risers will be checked against the high port velocity runs. The better of the two type ports will be used for the horizontal pipes.

    Tests will be made with both single and double level horizontal distributors. The liner will remain in both the retorting and combustion zones for these studies.

11. Retort No. 1 was repaired and put into service. The warped center section was removed. External stiffeners were installed at the combustion zone and supports were mounted at the top of the unit to prevent slumping and bulging during periods of very high temperatures.

    The center air port was lowered and is now two inches above the peripheral inlets.

    Orifices were installed on the lines to the center and peripheral air distributors to regulate air or gas splits.

    A double star feeder was installed at the spent shale discharge to prevent gas leakage at this point.

    Facilities to supply, measure, and regulate oxygen flow were provided for the high Cp studies.

    The top bed height capability was raised from seven to nine feet.

    The retort was completely reinsulated. Additional pressure taps were installed.

12. Further design work has been initiated for Retort No. 1, to incorporate three additional peripheral inlet sections 12-inches apart above the existing inlet. Hot inert gas addition will be tested at various levels.

13. The crushing plant was shut down August 12 for inspection. The Research Foundation reported that three days would be required to repair the bearings found damaged on the primary jaw crusher. Ten days were required.

    Three main bearings were eventually rebabbitted. However, the main problem was with cracks in the Pitman water cooled bearing. The Research Foundation shipped all bearings to Grand Junction for brazing and babbitting.

Several of the babbitt jobs were poor and required rebabbitting. Then, additional cracks developed in the Pitman Cap. At this point, the Research Foundation requested our advice. The Traylor Company was contacted and their procedures for rewelding cracks, rebabbitting, plus shaft clearances obtained. The Research Foundation completed their work using Traylor instructions Monday morning, August 23.

IV. RETORTING SECTION (J. E. Lawson)

A. Retorting Group (R. L. Clampitt, D. P. Cotrupe, P. H. Gifford, J. W. Hasz, K. I. Jagel and E. E. Turner

Summary

Most of this month has been spent operating Retort No. 2. The last few days have been spent shaking down and operating Retort No. 1 after a long down period.

The operability of Retort No. 2 has been improved considerably by three recent modifications. The effect of using a riser type distributor was shown to improve operability in last month's report. Since then we have tested the effect of a stainless steel liner installed in the retort and repaired a loose roller on the roll feeder. The installation of the liner has particularly improved operability.

Yields in the 87% range have been obtained on Retort No. 2 at 500 lb/(hr)(ft^2) raw shale at relatively high recycle rates and low air rates. An attempt to reproduce Run 507, 508 and 509 yields obtained in Retort No. 1 has been unsuccessful.

A new technique of sampling shale from the moving shale bed has been developed. Dust concentration in the retorting and combustion zones is as high as 30 to 50 times what it is in the raw shale feed.

A new jacketed gas sampling probe has permitted us to improve the quality of our gas sampling within the retort during operation. There are still some mechanical problems with the sampler, however, it can be tentatively concluded that oxygen concentration in the 2 to 5% range exist six inches above the air inlets.

A new method of evaluating the uniformity of shale movement during retort operation has also been developed. Steel rings are inserted around the periphery and in the center of the retort and are picked up from the spent shale belt by an electromagnet. The time of travel is measured.

The dust carried out by the spent shale rotoclone is a significant, but small, stream of organic carbon leaving the unit which is unaccounted for. In one run, if this were taken into consideration the organic carbon balance was increased by about 0.5%. Some work is also being done to improve the sampling of liquid product and the analysis of the liquid product for water. This sampling and analysis affects both yield and organic carbon balance.

A slow equilibration of the unit after startup or after a process change has been shown to exist. This line out is shown by

gradual increases in the organic carbon content of both the gas and spent shale. It appears to be consistent with the accumulation of an organic carbon rich material inside the retort. Tentatively, we have concluded that this is the dust which has been shown to exist in the retorting and combustion zones.

1. Retort No. 2 Operations

a. Effect of Hardware Changes on Operability

During the vacation shutdown, the major modification made to the retort was the installation of a stainless steel liner. The liner was installed to provide a wall surface similar to that in Retort No. 1. Operation to date indicates that the liner has had a definite and beneficial effect. Bed height requirements previously determined on Retort No. 1 now apply to the Retort No. 2. Before installation of the liner, bed height requirement for a given offgas temperature was about 2-feet greater than required on the No. 1 or lined No. 2 Retort.

It is fairly certain that the change in operation of the retort resulted from the elimination of stable shale bridge above the air distributors. Installation of the liner provided a slick wall surface and eliminated anchor points necessary to support and maintain the bridge. It should be emphasized that the bridge was not static - it did not prevent shale flow - but was a dynamic, steady-state bridge through which the shale moved. Thief samples taken with a 3-inch pipe through the wall of the retort immediately above the air distributor bayonets during Run B665 (prior to the vacation shutdown) showed an extremely low bed density above the distributors. The thief samples obtained consisted primarily of dust with few large pieces of shale included. The bed also offered no resistance to probing. Subsequent thiefing of the bed since installation of the liner shows a high density bed composed of large shale particles and dust. A large resistance to probing is also noted.

The change in bed height requirement and the probing experience indicates that prior to the installation of the liner a low density expanded bed about 2-feet in height existed in the region of the air distributors. This bed configuration can account for a number of the operating difficulties experienced on the Retort No. 2 such as: (1)difficulty in controlling the shale level in the standpipe; (2)the observation that occasionally something "broke loose" in the retort; and (3)the formation of clinkers at high levels in the retort.

During the shutdown necessitated by the crusher failure, it was found that the east roll feeder at the bottom of the retort had broken loose from its drive shaft. Attempts have been made to determine how long the roll had been broken. However, the various observations that can be made are over shadowed to a large degree by the effect of the stainless steel liner. About the only conclusions that can be reached are:

(1) A review of Gamm-O-Tron charts indicates that the roll may have broken between Runs B445 and B446, last December.

(2) The broken roll probably contributed materially to the startup difficulties experienced on the retort.

(3) Mechanical model studies indicate that once shale flow is properly established, the rolls should not have much effect at the level of the air distributors.

The unit was started up without difficulty on August 23. Operation to date has been smooth and continuous. Although 90% FA yields have not yet been obtained, the objective of providing an operable retort has been achieved.

b. Effect of Process Variables

Reasonable operability was achieved after the riser type air distributor was installed. (This was done immediately prior to Run B659.) Because of low organic carbon balances, short run time, or failure to initiate combustion at all eight of the risers, the first significant data were achieved in Run B674. This run as well as replicates of this run having organic carbon balances in the 96 to 104% range are presented in Table 1. This series of runs was carried out at about the same conditions as were used in Runs 507, 508 and 509 which were carried out in Retort No. 1. The air and recycle rates are lower by about 400 SCF/Ton RS and 1,000 SCF/Ton RS, respectively and the bed height is 2-feet higher. The average yield is about 5.6% lower than was achieved in Retort No. 1. (Currently, a study is being carried out in Retort No. 1 to determine if the yields of Runs 507, 508 and 509 are reproducible.) It is doubtful that this yield difference can be attributed to the differences in process conditions. An alternative explanation is that the retort hardware is responsible for the yield difference. A study of the effect of some of the hardware variables is planned in Retort No. 2. Some of these studies have been completed and more are planned. In addition, a very limited examination of process variable effects has been carried out with this retort and air distributor design. With these studies, we have considered this attempt at reproducing Runs 507, 508 and 509 as a base case.

TABLE 1

RETORT NO. 2 ATTEMPT TO REPRODUCE RUN 507-9 PERFORMANCE (BASE CASE)

RUN NUMBER	B674	B675	B678.	B679.	Base
DATE STARTED	8-16-65	8-16-65	8-25-65	8-25-65	Case
LENGTH OF RUN, hours	8	8	8	8	Avg.
RETORT TYPE NUMBER	RB III	RB III	RB III	RB III	
OIL RECOVERY SYSTEM NUMBER	B9	B9	B9	B9	
OPERATING CONDITIONS:					
Raw Shale, lbs/(hr)(ft^2)	508	506	520	525	515
Fischer Assay, Gal/Ton RS	24.6	25.4	26.9	26.6	25.9
Nom. Size Range, inches		3/4 - 1 1/2			
Avg. Part. Diam., inches	0.23	0.36	0.30	0.27	0.29
Air, SCF/Ton RS	4928	5118	4909	4771	4932
Tot. Recycle, SCF/Ton RS (wet)	11630	11780	11570	11400	11600
Dilution Gas, SCF/Ton RS (wet)	—	—	—	—	—
Propane, SCF/Ton RS	—	—	—	—	—
Brine, Gal/Ton RS	—	—	—	—	—
Air Temp. Entering Retort, °F	176	182	182	199	155
Bed Hgt. Above Air Dist., ft	9	9	9	9	9
AIR DISTRIBUTOR	VII	VII	VII	VII	VII
OPERATING DATA:					
Offgas Temperature, °F	132	134	132	133	133
Recycle Gas Temperature, °F	188	192	180	191	188
Spent Shale Temperature, °F (EST.)	(450)	(450)	384	423	427
Avg. Retort ΔP, in. H_2O/ft	0.33	0.27	0.51	0.47	0.40
ΔP Above Air Dist. in H_2O/ft	0.41	0.32	0.61	0.53	0.48
Overall Oper. Performance	GOOD	GOOD	GOOD	GOOD	Good
PRODUCTS RECOVERED:					
Oil Collected, vol % RSFA	81.9	84.5	83.3	84.3	8[illegible]
Oil Lost as Mist, vol % RSFA	0.3	0.4	0.4	0.3	0.4
Oil in Spent Shale, vol % RSFA	0.0	0.0	0.0	0.0	0.[illegible]
Total Oil Meas., vol % RSFA	82.2	84.9	83.7	84.6	[illegible].9
Total Water, lbs/Ton RS	68.5	74.5	86.4	76.8	76.6
Calc. Dry Vent Gas, SCF/Ton RS	6280	6340	6122	5876	6155
Mineral CO_2 Decomposed, %	30.3	30.6	36.0	33.6	32.6
MATERIAL BALANCES:					
Ash, wt % (measured)	97.8	98.6	96.6	93.7	96.[illegible]
Basis for Yields & Mat'l. Bal.	RS	RS	RS	RS	RS
Overall Balance, wt %	100.6	100.0	99.4	99.3	[illegible].6
Organic Carbon Balance, wt %	97.5	99.4	99.7	97.9	98.6
Total Carbon Balance, wt %	98.2	99.6	99.7	98.5	99.0
Organic Hydrogen Balance, wt %	103.1	98.6	104.8	101.1	[illegible]1.9
Water Balance, wt %	96.0	110.7	108.3	98.5	101.6
Gas Loss, SCF/Ton RS (dry)	-402	-358	344	-450	-35[illegible]
HEAT BALANCE:					
Heat of Combustion, MBtu/Ton RS	476.5	484.7	484.5	468.6	478.6
Unaccounted Heat, MBtu/Ton RS	60.1	70.0	83.9	63.8	69.5
SHALE OIL PROPERTIES:					
Gravity, °API	19.0	19.1	19.3	19.1	19.1
Ramsbottom Carbon, wt %	2.23	2.35	2.41	2.42	2.35
Ash, wt %	0.02	0.02	0.01	0.01	0.02
GAS PROPERTIES (DRY):					
Moisture, lbs/MSCF of dry gas	9.2	10.1	10.9	11.4	10.4
Gross Heating Value, Btu/SCF	107	111	130	120	117
O_2 vol %	0.2	0.4	0.3	0.6	0.4
CO_2 vol %	24.5	24.8	25.8	25.0	2[illegible]
SPENT SHALE:					
Fischer Assay, Gal/Ton SS	0.0	0.0	0.0	0.0	0.0
Organic Carbon, wt %	1.75	1.94	1.82	1.89	1.85

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(1) Effect of Bed Height (Other Variables at Base Case Conditions)

In Table 2, a comparison of the base case average results is made with Run B681. This comparison shows that there is no significant difference in yield. A vertical temperature profile carried out in Run B678 showed that the two top feet of the retort were isothermal. Therefore, the lack of a yield effect on bed height is not unexpected.

(2) Effect of Dilution Gas (Other Variables, Except Bed Height and Dilution Gas Rate, at Base Case Conditions)

The effect of dilution gas on retorting performance when other variables are held at base case conditions is shown in Table 3. There is probably no effect on yield indicated but there is evidence that the combustion zone is affected. The organic carbon content of the spent shale is higher in the dilution gas situation than it is in the base case. This probably indicates less coke burning. The carbonate decomposition is also lower in the dilution gas case indicating that the average temperature of the combustion zone is probably also reduced.

(3) Effect of Increased Gas Throughput (Recycle Rate was Increased and Air Rate was Decreased, Other Variables at Base Case Conditions)

Increased gas throughput accompanied with a slightly increased heat input to the combustion zone causes a slight increase in yield. This is shown in Table 4. The fact that heat input to the combustion zone was increased was inferred from the increased carbonate decomposition. That part of this heat came from improved heat transfer in the shale cooling zone is shown by the low spent shale temperature.

(4) Effect of Dilution Gas at High Gas Throughput (Condition Same as 3 Above Except Dilution Gas Added)

Runs B693 and B694 were made at these conditions but no firm conclusions can be drawn as to yield because of poor organic carbon balances. The indicated spent shale temperatures are about the same as above as might be expected. The organic carbon content in the spent shale was increased and the percent carbonate decomposed was reduced. These observations are consistent with the conclusions drawn as to the effect of dilution gas previously. This is shown in Table 5.

TABLE 2

EFFECT OF BED HEIGHT (OTHER VARIABLES AT BASE CASE CONDITIONS)

RUN NUMBER	B681			BASE	
DATE STARTED	8-25-65			CASE	
LENGTH OF RUN, hours	8			AVG	
RETORT TYPE NUMBER	RBIII			(SEE	
OIL RECOVERY SYSTEM NUMBER	89			TABLE 1.)	
OPERATING CONDITIONS:					
Raw Shale, lbs/(hr)(ft^2)	516			515	
Fischer Assay, Gal/Ton RS	26.7			25.9	
Nom. Size Range, inches	3/4 -	1½			
Avg. Part. Diam., inches				0.29	
Air, SCF/Ton RS	4843			4932	
Tot. Recycle, SCF/Ton RS (wet)	11590			11600	
Dilution Gas, SCF/Ton RS (wet)	–			–	
Propane, SCF/Ton RS	–			–	
Brine, Gal/Ton RS	–			–	
Air Temp. Entering Retort, °F	202			185	
Bed Hgt. Above Air Dist., ft	7			9	
AIR DISTRIBUTOR	VII			VI	
OPERATING DATA:					
Offgas Temperature, °F	135			132	
Recycle Gas Temperature, °F	180			188	
Spent Shale Temperature, °F	429			427	
Avg. Retort ΔP, in. H_2O/ft	0.48			0.40	
ΔP Above Air Dist. in H_2O/ft	0.55			0.48	
Overall Oper. Performance	GOOD			GOOD	
PRODUCTS RECOVERED:					
Oil Collected, vol % RSFA	81.3			83.5	
Oil Lost as Mist, vol % RSFA	0.3			0.4	
Oil in Spent Shale, vol % RSFA	0.0			0.0	
Total Oil Meas., vol % RSFA	81.6			83.9	
Total Water, lbs/Ton RS	81.3			76.6	
Calc. Dry Vent Gas, SCF/Ton RS	6127			6155	
Mineral CO_2 Decomposed, %	33.4			32.6	
MATERIAL BALANCES:					
Ash, wt % (measured)	94.6			96.8	
Basis for Yields & Mat'l. Bal.	RS			RS	
Overall Balance, wt %	100.1			99.6	
Organic Carbon Balance, wt %	96.3			98.6	
Total Carbon Balance, wt %	97.4			99.0	
Organic Hydrogen Balance, wt %	101.3			101.9	
Water Balance, wt %	115.7			101.6	
Gas Loss, SCF/Ton RS (dry)	403			-389	
HEAT BALANCE:					
Heat of Combustion, MBtu/Ton RS	475.7			478.6	
Unaccounted Heat, MBtu/Ton RS	53.0			69.5	
SHALE OIL PROPERTIES:					
Gravity, °API	19.0			19.1	
Ramsbottom Carbon, wt %	2.58			2.35	
Ash, wt %	0.09			0.02	
GAS PROPERTIES (DRY):					
Moisture, lbs/MSCF of dry gas	11.7			10.4	
Gross Heating Value, Btu/SCF	114			117	
O_2 vol %	0.2			0.4	
CO_2 vol %	25.7			25.0	
SPENT SHALE:					
Fischer Assay, Gal/Ton SS	0.0			0.0	
Organic Carbon, wt %	1.81			1.85	

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TABLE 3

EFFECT OF DILUTION GAS (OTHER VARIABLES, EXCEPT BED HEIGHT AND DILUTION GAS RATE, AT BASE CASE CONDITIONS)

RUN NUMBER	B683	B684	B685	AVG.	BASE
DATE STARTED	8-27-65	8-28-65	8-29-65		CASE
LENGTH OF RUN, hours	8	8	8		AVG
RETORT TYPE NUMBER	RB III	RB III	RB III		(SEE
OIL RECOVERY SYSTEM NUMBER	89	89	89		TABLE 1)
OPERATING CONDITIONS:					
Raw Shale, lbs/(hr)(ft^2)	505	510	518	511	515
Fischer Assay, Gal/Ton RS	26.4	27.0	26.5	26.6	25.9
Nom. Size Range, inches	3/4 - 1½				
Avg. Part. Diam., inches					0.29
Air, SCF/Ton RS	4923	4897	4814	4878	4932
Tot. Recycle, SCF/Ton RS (wet)	12876	12946	12837	12886	11600
Dilution Gas, SCF/Ton RS (wet)	1048	1046	1037	1044	—
Propane, SCF/Ton RS	—	—	—	—	—
Brine, Gal/Ton RS	—	—	—	—	—
Air Temp. Entering Retort, °F	208	203	203	205	185
Bed Hgt. Above Air Dist., ft	7	7	7	7	9
Air Distributor	VII	VII	VII	VII	VII
OPERATING DATA:					
Offgas Temperature, °F	135	134	135	135	133
Recycle Gas Temperature, °F	193	196	198	196	188
Spent Shale Temperature, °F	474	435	443	447	427
Avg. Retort ΔP, in. H_2O/ft	0.48	0.54	0.51	0.51	0.40
ΔP Above Air Dist. in H_2O/ft	0.56	0.69	0.62	0.62	0.48
Overall Oper. Performance	GOOD	GOOD	GOOD	GOOD	GOOD
PRODUCTS RECOVERED:					
Oil Collected, vol % RSFA	81.9	89.5	83.5	84.9	83.5
Oil Lost as Mist, vol % RSFA	0.3	0.3	0.3	0.3	0.4
Oil in Spent Shale, vol % RSFA	0.0	2.4	0.0	0.8	0.0
Total Oil Meas., vol % RSFA	82.2	92.2	83.8	86.0	83.9
Total Water, lbs/Ton RS	80.0	75.0	76.6	77.2	76.6
Calc. Dry Vent Gas, SCF/Ton RS	6110	6117	5985	6071	6155
Mineral CO_2 Decomposed, %	28.8	28.1	29.7	28.9	32.6
MATERIAL BALANCES:					
Ash, wt % (measured)	93.8	91.0	94.7	93.2	96.8
Basis for Yields & Mat'l. Bal.	RS	RS	RS	RS	RS
Overall Balance, wt %	100.6	101.0	100.1	100.6	99.6
Organic Carbon Balance, wt %	98.6	103.5	97.9	100.0	98.6
Total Carbon Balance, wt %	99.0	102.5	98.5	100.0	99.0
Organic Hydrogen Balance, wt %	101.3	100.3	99.8	100.5	101.9
Water Balance, wt %	124.0	127.5	117.6	123.0	101.6
Gas Loss, SCF/Ton RS (dry)	815	-32	-574	452	-389
HEAT BALANCE:					
Heat of Combustion, MBtu/Ton RS	470.6	465.8	468.6	468.3	478.6
Unaccounted Heat, MBtu/Ton RS	48.8	76.9	58.0	61.2	69.5
SHALE OIL PROPERTIES:					
Gravity, °API	19.4	19.3	19.3	19.3	19.1
Ramsbottom Carbon, wt %	2.04	2.23	2.13	2.13	2.35
Ash, wt %	0.03	0.02	0.02	0.02	0.02
GAS PROPERTIES (DRY):					
Moisture, lbs/MSCF of dry gas	10.4	9.0	10.1	9.8	10.4
Gross Heating Value, Btu/SCF	110	116	106	111	117
O_2 vol %	0.4	0.2	0.2	0.3	0.4
CO_2 vol %	24.9	25.3	25.2	25.1	25.0
SPENT SHALE:					
Fischer Assay, Gal/Ton SS	0.0	0.3	0.0	0.1	0.0
Organic Carbon, wt %	2.28	2.47	1.96	2.23	1.85

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TABLE 4

EFFECT OF GAS THROUGHPUT (RECYCLE RATE INCREASED, AIR RATE DECREASED, OTHER VARIABLES AT BASE CASE CONDITIONS)

RUN NUMBER	B687	B688	Avg.	Base
DATE STARTED	8-30-65	8-31-65		Case
LENGTH OF RUN, hours	8	8		Avg.
RETORT TYPE NUMBER	RBIII	RBIII		(See
OIL RECOVERY SYSTEM NUMBER	B9	B9		Table 1.)
OPERATING CONDITIONS:				
Raw Shale, lbs/(hr)(ft^2)	517	514	516	515
Fischer Assay, Gal/Ton RS	27.9	27.9	27.9	25.9
Nom. Size Range, inches	3/4 - 1 1/2			
Avg. Part. Diam., inches				0.79
Air, SCF/Ton RS	4340	4335	4338	4632
Tot. Recycle, SCF/Ton RS (wet)	15880	16030	15960	11600
Dilution Gas, SCF/Ton RS (wet)	—	—	—	—
Propane, SCF/Ton RS	—	—	—	—
Brine, Gal/Ton RS	—	—	—	—
Air Temp. Entering Retort, °F	205	205	205	185
Bed Hgt. Above Air Dist., ft	9	9	9	9
Air Distributor	VII	VII	VII	VII
OPERATING DATA:				
Offgas Temperature, °F	137	137	137	133
Recycle Gas Temperature, °F	185	185	185	158
Spent Shale Temperature, °F	305	305	305	427
Avg. Retort ΔP, in. H_2O/ft	1.01	1.01	1.01	0.40
ΔP Above Air Dist. in H_2O/ft	1.35	1.35	1.35	0.48
Overall Oper. Performance	Good	Good	Good	Good
PRODUCTS RECOVERED:				
Oil Collected, vol % RSFA	86.5	87.1	86.8	83.5
Oil Lost as Mist, vol % RSFA	0.3	0.3	0.3	0.4
Oil in Spent Shale, vol % RSFA	0.0	0.0	0.0	0.0
Total Oil Meas., vol % RSFA	86.8	87.4	87.1	83.9
Total Water, lbs/Ton RS	70.7	72.2	71.5	76.6
Calc. Dry Vent Gas, SCF/Ton RS	5742	5769	5656	6155
Mineral CO_2 Decomposed, %	34.9	37.8	36.4	32.6
MATERIAL BALANCES:				
Ash, wt % (measured)	95.2	95.8	95.5	96.8
Basis for Yields & Mat'l. Bal.	RS	RS	RS	RS
Overall Balance, wt %	100.0	99.7	99.9	99.6
Organic Carbon Balance, wt %	97.6	96.0	96.8	98.6
Total Carbon Balance, wt %	98.3	97.1	97.7	99.0
Organic Hydrogen Balance, wt %	101.2	102.4	101.8	101.9
Water Balance, wt %	99.9	100.6	100.2	101.6
Gas Loss, SCF/Ton RS (dry)		1610		-389
HEAT BALANCE:				
Heat of Combustion, MBtu/Ton RS	429.5	433.6	431.6	478.6
Unaccounted Heat, MBtu/Ton RS	37.2	24.2	30.7	69.5
SHALE OIL PROPERTIES:				
Gravity, °API	19.5	19.7	19.6	19.1
Ramsbottom Carbon, wt %	2.16	2.28	2.22	2.35
Ash, wt %	0.02	0.04	0.03	0.02
GAS PROPERTIES (DRY):				
Moisture, lbs/MSCF of dry gas	10.0	10.0	10.0	10.4
Gross Heating Value, Btu/SCF	109	114	112	117
O_2 vol %	0.1	0.0	0.1	0.4
CO_2 vol %	26.7	27.8	27.3	25.0
SPENT SHALE:				
Fischer Assay, Gal/Ton SS	0.0	0.0	0.0	0.0
Organic Carbon, wt %	2.06	2.09	2.08	1.85

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TABLE 5

EFFECT OF DILUTION GAS AT HIGH GAS (CONDITION SAME AS PREVIOUS TABLE EXCEPT DILUTION GAS ADDED)

RUN NUMBER	[illegible]	[illegible]04	Avg.	[illegible]	[illegible]
DATE STARTED	[illegible]-2-65	9-[illegible]		[illegible]	
LENGTH OF RUN, hours	8	8		[illegible]	[illegible]
RETORT TYPE NUMBER	[illegible]	[illegible]		[illegible] Table	[illegible]
OIL RECOVERY SYSTEM NUMBER	[illegible]	64		4.)	Table 1.
OPERATING CONDITIONS:					
Raw Shale, lbs/(hr)(ft^2)	[illegible]	513	[illegible]	516	[illegible]
Fischer Assay, Gal/Ton RS	[illegible]	31.8	[illegible]	27.9	25.[illegible]
Nom. Size Range, inches	<	3/4	1½	—	—
Avg. Part. Diam., inches	[illegible]	0.29	0.29		0.29
Air, SCF/Ton RS	4236	4198	[illegible]217	4332	4582
Tot. Recycle, SCF/Ton RS(wet)	[illegible]	[illegible]	[illegible]169	1569[illegible]	11600
Dilution Gas, SCF/Ton RS (wet)	1020	[illegible]	1013	—	—
Propane, SCF/Ton RS	—	—	—	—	—
Brine, Gal/Ton RS	—	—	—	—	—
Air Temp. Entering Retort, °F	182	175	[illegible]	205	185
Bed Hgt. Above Air Dist., ft	9	9	9	9	9
Air Distributor	VII	VII	VII	VII	VII
OPERATING DATA:					
Offgas Temperature, °F	138	141	140	137	133
Recycle Gas Temperature, °F	192	187	189	185	185
Spent Shale Temperature, °F	335	285	310	305	427
Avg. Retort ΔP, in. H_2O/ft	0.92	[illegible]	0.93	1.01	0.4[illegible]
ΔP Above Air Dist. in H_2O/ft	0.59	[illegible]	0.61	1.35	0.48
Overall Oper. Performance	Good	Good	Good	Good	Good
PRODUCTS RECOVERED:					
Oil Collected, vol % RSFA	88.2	85.5	86.9	86.8	83.5
Oil Lost as Mist, vol % RSFA	0.3	0.3	0.3	0.3	0.4
Oil in Spent Shale, vol % RSFA	0.0	0.0	0.0	0.0	0.0
Total Oil Meas., vol % RSFA	88.5	85.8	87.2	87.1	83.9
Total Water, lbs/Ton RS	79.9	79.5	79.7	71.5	76.6
Calc. Dry Vent Gas, SCF/Ton RS	[illegible]	[illegible]	5404	5656	6155
Mineral CO_2 Decomposed, %	31.8	31.0	31.4	36.4	32.6
MATERIAL BALANCES:					
Ash, wt % (measured)	97.1	93.3	95.2	95.5	96.8
Basis for Yields & Mat'l. Bal.	RS	RS	RS	RS	RS
Overall Balance, wt %	99.5	99.5	99.5	99.9	99.6
Organic Carbon Balance, wt %	94.9	93.4	94.2	96.8	98.6
Total Carbon Balance, wt %	96.3	95.1	95.7	97.7	99.0
Organic Hydrogen Balance, wt %	103.9	100.3	102.1	101.8	101.9
Water Balance, wt %	108.0	104.9	106.5	100.2	101.6
Gas Loss, SCF/Ton RS (dry)	1410	1428	1419		-389
HEAT BALANCE:					
Heat of Combustion, MBtu/Ton RS	423.7	417.4	420.6	431.6	478.6
Unaccounted Heat, MBtu/Ton RS	22.2	22.2	22.2	30.7	69.5
SHALE OIL PROPERTIES:					
Gravity, °API	19.2	19.2	19.2	19.6	19.1
Ramsbottom Carbon, wt %	2.17	2.26	2.22	2.22	2.35
Ash, wt %	0.01	0.02	0.02	0.03	0.02
GAS PROPERTIES (DRY):					
Moisture, lbs/MSCF of dry gas	10.2	9.7	10.0	10.0	11.4
Gross Heating Value, Btu/SCF	117	[illegible]	125	112	117
O_2 vol %	0.1	0.1	[illegible]	0.1	[illegible]
CO_2 vol %	26.1	26.[illegible]	26.1	27.3	25.0
SPENT SHALE:					
Fischer Assay, Gal/Ton SS	0.0	0.0	0.0	0.0	0.0
Organic Carbon, wt %	2.25	2.37	2.31	2.08	1.[illegible]

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(5) Effect of Operation at Conditions Approaching Minimum Operable Heat Input

A pair of runs were also made at 400 lb/(hr)(ft^2) which approached the condition of minimum operable heat input (Runs B696 and B697). One of these runs had an acceptable organic carbon balance. The yield was about the same as the high gas throughput run at 500 lb/(hr)(ft^2). The percent carbonate decomposed was much lower and the organic carbon content of the spent shale was slightly greater than at the 500 lb/(hr)(ft^2) conditions. This is shown in Table 6.

(6) Reproducibility of Base Case Operation

After completing Run B697, an attempt was made to reproduce the base case operation. It was not possible to do this at that time. This is shown in Table 7. The retort was shut down two runs after this attempt at reproducing the base case data. An inspection of the retort indicated that three of the four bayonets on the west side were plugged with soft deposits in each of the ports and two of the four on the east side were partially plugged. A clinker was also observed resting on top of the riser in the southwest corner of the retort. The clinker was burned and somewhat glassy in the bottom indicating that it was there during some portion of the retort's operation. The top of the clinker had the appearance of coked oil. Since the top of this clinker was in what would normally be the bottom of the retorting zone or the top of the combustion zone, it suggests that this clinker may have fallen as a dusty, oily shale agglomerate into this position from the region of the retort normally containing oil.

The temperature of the streams leaving the retort are about the same for the new data as for the base case although the spent shale temperature may be slightly lower due to the slightly higher recycle.

The Ramsbottom carbon is 50% higher in the new data liquid product than in the base case. This probably indicates a greater propensity for the oil to coke and release light cracked products. The facts that the heating value of the gas and the organic carbon on the spent shale are higher are consistent with this. If these light cracked products were in the troublesome light naphtha region and if they were not recovered as part of the gas sample in a representative proportion the poor carbon balance of these data would also be accounted for.

TABLE 6

EFFECT OF OPERATION AT CONDITIONS APPROACHING MINIMUM OPERABLE HEAT INPUT

RUN NUMBER	R697		[illegible]		[illegible]
DATE STARTED	9-6-65		[illegible]		[illegible]
LENGTH OF RUN, hours	8		[illegible]		[illegible]
RETORT TYPE NUMBER	[illegible]		[illegible] Table		[illegible] Table
OIL RECOVERY SYSTEM NUMBER	89		[illegible]		[illegible]
OPERATING CONDITIONS:					
Raw Shale, lbs/(hr)(ft^2)	395		516		515
Fischer Assay, Gal/Ton RS	30.2		27.9		25.9
Nom. Size Range, inches	—	3/4 - 1 1/2	—	—	—
Avg. Part. Diam., inches	0.30				0.29
Air, SCF/Ton RS	3488		4838		[illegible]
Tot. Recycle, SCF/Ton RS (wet)	19299		15690		[illegible]
Dilution Gas, SCF/Ton RS (wet)	—		—		—
Propane, SCF/Ton RS	—		—		—
Brine, Gal/Ton RS	—		—		—
Air Temp. Entering Retort, °F	183		205		105
Bed Hgt. Above Air Dist., ft	7		9		9
[illegible]	VII		VII		VII
OPERATING DATA:					
Offgas Temperature, °F	145		137		133
Recycle Gas Temperature, °F	192		185		128
Spent Shale Temperature, °F	245		325		[illegible]
Avg. Retort ΔP, in. H20/ft	0.63		1.01		0.40
ΔP Above Air Dist. in H20/ft	0.40		1.35		[illegible]
Overall Oper. Performance	Good		Good		Good
PRODUCTS RECOVERED:					
Oil Collected, vol % RSFA	86.8		86.8		83.5
Oil Lost as Mist, vol % RSFA	0.5		[illegible]		0.4
Oil in Spent Shale, vol % RSFA	0.0		0.0		0.0
Total Oil Meas., vol % RSFA	87.3		87.1		83.9
Total Water, lbs/Ton RS	71.1		71.5		76.6
Calc. Dry Vent Gas, SCF/Ton RS	4505		5656		6155
Mineral CO2 Decomposed, %	20.3		36.4		32.6
MATERIAL BALANCES:					
Ash, wt % (measured)	95.0		95.5		96.8
Basis for Yields & Mat'l. Bal.	RS		RS		RS
Overall Balance, wt %	99.9		99.9		99.6
Organic Carbon Balance, wt %	96.9		96.8		[illegible]
Total Carbon Balance, wt %	97.7		97.7		99.0
Organic Hydrogen Balance, wt %	96.3		101.8		[illegible]
Water Balance, wt %	127.0		100.2		101.6
Gas Loss, SCF/Ton RS (dry)	1403				[illegible]
HEAT BALANCE:					
Heat of Combustion, MBtu/Ton RS	332.8		431.6		[illegible]
Unaccounted Heat, MBtu/Ton RS	26.0		30.7		[illegible]
SHALE OIL PROPERTIES:					
Gravity, °API	20.1		19.6		19.1
Ramsbottom Carbon, wt %	2.30		112		[illegible]
Ash, wt %	0.05		0.1		[illegible]
GAS PROPERTIES (DRY):					
Mol. Wt., lbs/MSCF of dry gas	10.4		10.0		[illegible]
Gross Heating Value, Btu/SCF	114		112		[illegible]
O_2 vol %	0.1		0.1		0.0
CO_2 vol %	25.5		27.3		[illegible]
SPENT SHALE:					
Fischer Assay, Gal/Ton SS	0.0		0.0		0.0
Organic Carbon, wt %	2.36		2.08		1.85

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TABLE 7

REPRODUCIBILITY OF BASE CONDITIONS

RUN NUMBER	[illegible]	[illegible]	[illegible]		[illegible]
DATE STARTED	9-7-65	9-7-65	[illegible]		[illegible]
LENGTH OF RUN, hours	8	8	7		[illegible]
RETORT TYPE NUMBER	[illegible]	[illegible]	[illegible]		(See
OIL RECOVERY SYSTEM NUMBER	89	89	89		Table 1.)
OPERATING CONDITIONS:					
Raw Shale, lbs/(hr)(ft^2)	500	502	500		515
Fischer Assay, Gal/Ton RS	33.3	33.0	33.0		25.9
Nom. Size Range, inches	←	3/4 - 1 1/2"	→		→
Avg. Part. Diam., inches	0.37	0.35	0.35		0.29
Air, SCF/Ton RS	[illegible]	[illegible]	4870		[illegible]
Tot. Recycle, SCF/Ton RS (wet)	[illegible]	12072	12070		[illegible]
Dilution Gas, SCF/Ton RS (wet)	—	—	—		—
Propane, SCF/Ton RS	—	—	—		—
Brine, Gal/Ton RS	—	—	—		—
Air Temp. Entering Retort, °F	201	199	192		185
Bed Hgt. Above Air Dist., ft	7	7	7		9
[illegible]	VII	VII	VII		VII
OPERATING DATA:					
Offgas Temperature, °F	136	134	134		133
Recycle Gas Temperature, °F	199	196	192		182
Spent Shale Temperature, °F	375	413	418		427
Avg. Retort ΔP, in. H_2O/ft	0.44	0.45	0.49		0.42
ΔP Above Air Dist. in H_2O/ft	0.15	0.24	0.26		0.48
Overall Oper. Performance	Good	Good	Good		Good
PRODUCTS RECOVERED:					
Oil Collected, vol % RSFA	79.6	77.8	78.4		83.5
Oil Lost as Mist, vol % RSFA	0.4	0.4	0.4		0.4
Oil in Spent Shale, vol % RSFA	0.0	0.0	0.0		0.0
Total Oil Meas., vol % RSFA	80.0	78.2	78.8		83.9
Total Water, lbs/Ton RS	72.8	76.4	73.7		76.6
Calc. Dry Vent Gas, SCF/Ton RS	[illegible]	6216	6302		[illegible]
Mineral CO_2 Decomposed, %	31.2	30.8	30.9		32.6
MATERIAL BALANCES:					
Ash, wt % (measured)	95.4	97.6	93.1		96.8
Basis for Yields & Mat'l. Bal.	RS	RS	RS		RS
Overall Balance, wt %	98.7	98.9	99.1		[illegible]
Organic Carbon Balance, wt %	94.9	91.1	90.9		[illegible]
Total Carbon Balance, wt %	96.1	[illegible]	93.1		[illegible]
Organic Hydrogen Balance, wt %	98.6	97.5	98.0		101.9
Water Balance, wt %	98.1	97.5	99.0		101.6
Gas Loss, SCF/Ton RS (dry)	-185	-297	-144		-389
HEAT BALANCE:					
Heat of Combustion, MBtu/Ton RS	446.9	458.9	[illegible]		[illegible]
Unaccounted Heat, MBtu/Ton RS	45.3	35.4	22.7		[illegible]
SHALE OIL PROPERTIES:					
Gravity, °API	18.7	19.0	18.9		19.1
Ramsbottom Carbon, wt %	3.08	3.24	3.10		[illegible]
Ash, wt %	0.03	0.13	0.04		0.02
GAS PROPERTIES (DRY):					
Moisture, lbs/MSCF of dry gas	9.8	9.9	9.4		10.4
Gross Heating Value, Btu/SCF	154	151	[illegible]		[illegible]
O_2 vol %	[illegible]	[illegible]	[illegible]		[illegible]
CO_2 vol %	22.7	22.8	23.1		[illegible]
SPENT SHALE:					
Fischer Assay, Gal/Ton SS	6.0	[illegible]	6.0		6.0
Organic Carbon, wt %	2.78	2.53	2.57		[illegible]

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This change in the properties of streams leaving the retort may be due to a change in shale richness, but after examining the unit, it seems more probable that it is due to changes in shale and gas flow distribution due to port plugging and clinker formation.

c. Shale Flow Studies During Hot Operation

Uniform shale flow through a retort may be important in obtaining good operability and yields. Temperature profiles, residence time, fines refluxing, gas distribution and other variables could be affected by non-uniform shale flow. Tracer tests have been conducted in Retort No. 2 to determine the flow pattern of the shale. The tests were made by dropping metal cylinders, cut from pipe, into the top of the retort (See Table 8) and catching the cylinders with a magnet placed over the belt after it leaves the retort. The travel time of the cylinder from the top of the retort to the magnet was obtained. To get the travel pattern in the middle of the bed, the cylinders were dropped through the chute feeding raw shale to the retort. The results of these tests are given in Table 8 and pertinent data are summarized below:

SUMMARY OF SHALE FLOW TESTS

Test No.	Date	Location in Retort	Average Residence Time (Mins)	Residence Time (Mins) Min.	Max.	Difference
1	8/27	S	107	101	120	19
2	8/27	N	99	98	99	1
3	8/30	S	120	110	127	17
4	8/30	N	164	105	233	123
5	8/30	N	115	106	125	19
		S	115	107	123	16
6	8/31	N	116	103	138	35
		S	117	107	123	16
7	8/31	E	121	112	134	22
		W	121	105	157	52
8	9/1	Chute	110	100	115	15
9	9/1	Chute	110	106	112	6
10	9/9	N	98	95	100	5
		S	130	112	143	31
		E	114	99	143	44
		W	108	92	134	42

On an average these data indicate a fairly uniform flow except for in a few instances. Test 4 indicates some variations in shale flow while tests taken about three hours later showed improved flow characteristics. Qualitatively, these limited data indicate somewhat faster

Average (Minutes)				
N	S	E	W	CHUTE
--	107	--	--	--
99	--	--	--	--
--	120	--	--	--
164	--	--	--	--
115	115	--	--	--
116	117	--	--	--
--	--	121	121	--
--	--	--	--	110
--	--	--	--	110
98	130	114	108	--

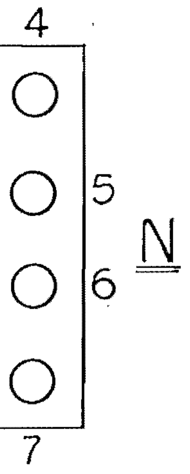

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TABLE 8

RESIDENCE TIME STUDIES - RETORT NO. 2

August 27 through September 9, 1965
7 Feet Above Air Inlet
500 lb/(hr)(ft^2) shale rate

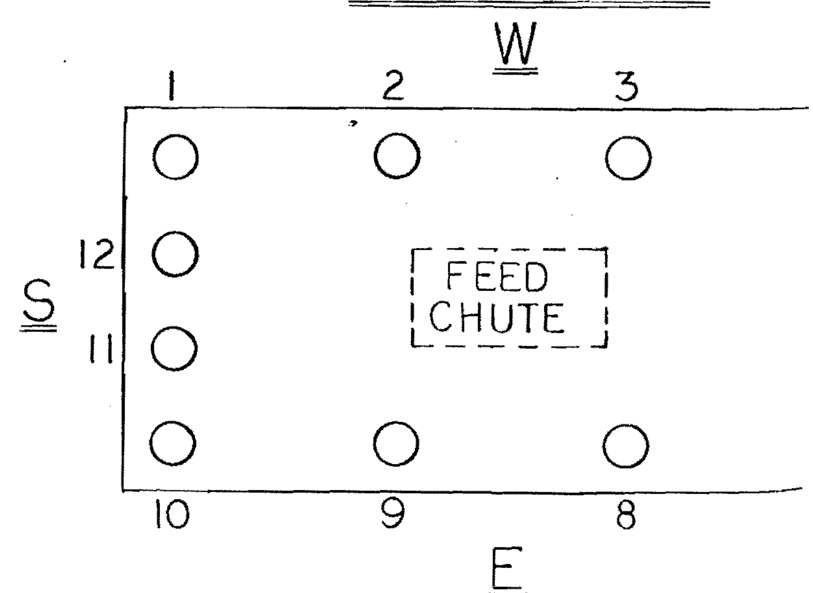
Test No.	Time	Date	Run No.	Drop Ports (Time in Minutes) Top of Retort (Numbers - See Below) 1	2	3	4	5	6	7	8	9	10	11	12	Feed Chute (*) A	B	C	D
1	--	8/27	B682	101	--	--	--	--	--	--	--	--	102	105	120	--	--	--	--
2	1600	8/27	B683	--	--	--	NR	98	99	100	--	--	--	--	--	--	--	--	--
3	0835	8/30	B686	117	--	--	--	---	--	--	--	--	110	124	127	--	--	--	--
4	1050	8/30	B686	--	--	--	105	215	105	233	--	--	--	--	--	--	--	--	--
5	1320	8/30	B687	110	--	--	NR	106	115	125	--	--	123	120	107	--	--	--	--
6	0905	8/31	B688	113	--	--	103	106	111	138	--	--	123	125	107	--	--	--	--
7	1135	8/31	B688	117	107	157	105	--	--	112	113	134	118	--	--	--	--	--	--
8	1050	9/1	B691	--	--	--	--	--	--	--	--	--	--	--	--	115	100	112	113
9	1325	9/1	B691	--	--	--	--	--	--	--	--	--	--	--	--	106	109	110	112
10	--	9/9	After B702	134	92	99	NR	98	95	100	NR	99	143	112	131	--	--	--	--

*Does not include holdup time in chute (14 min)

Note: Settings on Rolls
Test 1 S - 2 2/16; N - 2 3/16
Others S - 2 4/16; N - 2 3/16

North (n) - Ports 4, 5, 6, 7
East (e) - Ports, 7, 8, 9, 10
South (s) - Ports 10, 11, 12, 1
West (w) - Ports 1, 2, 3, 4
Feedchute - Rings A, B, C, D,
NR - Rings were not recovered

TOP OF RETORT



rates in the middle of the bed (average time N 124; S 115; Middle 110), although the time may be within the precision of the data. The calculated residence time of the shale is about 118 minutes.

Test 10 was made after Run B702 and before the unit was shut down. The data indicate poor flow characteristics in the south, southeast and southwest areas of the retort. After shutdown of the unit, a clinker was found over the bayonet in the southwest corner and the ports on three of four of the west bayonets were plugged with soft deposits. Two of the east bayonets were also partially plugged. The clinker and the plugged ports must have occurred sometime after Run B691 (9/1/65) as Tests 7, 8 and 9 showed good flow characteristics. The poor flow characteristics may have affected the yield results for the runs made prior to the shutdown.

d. Sampling Shale From a Moving Shale Bed

Starting with Run B689 a new type of sample has been obtained. It is called a thief sample, and is taken by driving a 3-inch pipe into the moving shale bed at given locations. The purpose of this sample is to gain additional insight into what is occurring in a moving bed of retorting shale, and more specifically to determine what the main causes of yield loss are. Prior to the development of this sampling technique an isokinetic dust sample was taken to serve this purpose. The isokinetic dust sample technique has now been abandoned in favor of the more reproducible and correlatable thief sample method.

Some of the results which have been obtained are presented on Tables 9 and 10. The data on Table 9 show the reproducibility of this technique. The three samples of Test No. 1 were taken at Port No. 2 at half hour intervals. This port is located 8-inches above the air inlet in the west wall of Retort No. 2. Originally, it was thought that the first sample contained a high weight percent dust because of the procedure used, but subsequent data, given under Test No. 2 show that even with a corrected procedure and an hour sampling period the subsequent samples equilibrate to a dust concentration different than the first sample.

In Table 10 the vertical dust profile for a typical high dust operation and a typical low dust operation is given. Although a more complete analyses was obtained, only the weight percent of material larger than the pan fraction and weight percent on the pan are given. Pan is defined as any material larger than 0.093 inches in order to conform with the raw shale screen analysis which were typically 1 to 2 percent pan. In addition to these data, the Fischer

TABLE 9

THIEF SAMPLING TECHNIQUE FOR DUST CONCENTRATION -
REPRODUCIBILITY TEST

	Test No. 1			Test No. 2			
	Weight Percent						
Screen Size	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4
0.742	27.8	41.4	34.3	31.8	60.1	54.2	55.0
0.624	5.4	12.1	13.2	4.0	6.6	11.2	2.2
0.414	9.4	10.4	13.3	2.9	3.4	3.1	3.2
0.371	2.9	3.5	4.1	1.2	0.7	0.4	0.3
0.263	4.0	4.0	3.7	1.3	0.7	0.8	0.7
0.185	3.[illegible]	3.2	3.3	1.0	0.7	0.7	1.2
0.131	3.2	2.8	2.9	0.9	0.8	0.7	0.9
0.093	3.1	2.4	2.6	1.0	0.7	0.6	0.9
0.065	3.5	2.7	2.6	1.4	0.9	0.9	1.3
0.0328	7.5	5.1	5.5	8.6	3.5	4.2	5.4
Pan	30.1	12.4	14.5	45.9	21.9	23.2	28.9
Total Weight of Sample	1417	1318	1516	757	880	647	457

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TABLE 10

VERTICAL PROFILE OF DUST CONCENTRATION AND SHALE PROPERTIES

	Typical High Dust	Typical Low Dust
Run No.	699	694
Raw Shale	502	513
Air	4,830	4,200
Recycle	12,071	17,000
Dilution	0	1,000
Bed Height	78	7
Yield		87

	Typical High Dust				Typical Low Dust			
	RS	23" Above	8" Above	SS	RS	23" Above	8" Above	SS
FA	33.0	13.7	2.5	0.0	31.6	28.4	0.4	0.0
CO_2	16.8	15.0	17.5	14.8	17.1	16.2	19.2	14.9
Ash	65.3	74.2	77.8	83.1	66.0	67.8	77.9	83.3
Carbon	19.0	13.9	9.07	6.6	18.4	17.4	7.93	6.43
Hydrogen	2.07	1.01	0.41	0.18	2.0	1.74	0.25	0.18
Wt % > Pan	98.5	42.5	56.6	--	97.9	89.7	72.5	--
Pan < 0.09	1.5	57.5	43.4	--	2.1	10.3	27.5	--

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9/20/65

Assay, CO_2, ash, total carbon, ash total hydrogen are given for each sample and for the raw and spent shale. With this information one can follow these variables as the shale passes through the retorting and combustion zones. The external process variables are given at the top of the table.

The main preliminary conclusion which was drawn from these results is that the greater the concentration of dust accompanying the raw shale through the retorting zone the slower the rate of retorting. This implies that the dust tends to slow down the rate of heat transfer to the incoming shale particles; it also gives the mechanism by which partially retorted shale enters the combustion zone. To fully substantiate these conclusions additional data are required.

The dust concentration is related to both the air and recycle rates as shown in Table 11. Run B699 with an air rate of 4,830 SCF/Ton RS and a total recycle of 12,000 SCF/Ton RS gave 57.5 wt % dust at port No. 1. When the air was lowered to 4,200 SCF/Ton RS and the total recycle raised to 17,000 SCF/Ton the dust dropped to 10 wt % at the same position. Thus the total gas traffic, air plus recycle went from 16,900 to 21,200 SCF/Ton RS while the dust dropped from 57.5 to 10 wt %. In B697, a 400 mass rate run, the air was 3,500 SCF/Ton, the recycle 19,300 SCF/Ton giving a total of 22,800. The dust concentration at position 1 went up slightly to 15.5 wt %. The conclusion which was drawn is that at sufficiently high recycle rates, the dust concentration will increase with increasing recycle rate, but at total recycle rate of around 17,000 SCF/T or less the air rate is the variable which most strongly influences the dust concentration. The postulated mechanism is this: increasing air rate increases the jet velocity through the air port. The surface of the particles in the region of the jet become very hot and this combined with the increased force of the jet causes attrition of the surface. Once attrition starts the rate of oxygen consumption per unit volume in the region of the jet may rise rapidly. This theory will be studied during the next period of operation of Retort No. 2, by lowering the jet velocity at a known air rate - dust concentration condition and determining what effect, if any, this variable has on the dust concentration.

e. <u>Sampling of Gas From a Moving Shale Bed, Primarily For Oxygen</u>

A new design for the oxygen probe was used starting with Run B683. This design entailed the use of a cooling jacket around the 1/4-inch sample tube. The purpose was to cool the gas as soon as it entered the tube. Prior to this design the results indicated that a change in the gas composition

TABLE 11

DUST CONCENTRATION VERSUS EXTERNAL VARIABLE

Run No.	B699	B694	B697
Raw Shale	502	513	395
Air	4800	4200	3500
Total Recycle	12,000	17,000	19,300
Dilution	0	1,000	0
Air + Recycle	16,800	21,200	22,800
Wt % Dust at 23" Above Air Inlet	57.5	10.3	15.5

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9/20/65

was most likely occurring when the non-jacketed, hot probe was used. Tests with the new probe confirmed that this was in fact happening.

Two typical gas analyses are shown in Table 12. They represent typical high and low oxygen concentration gases with the bayonet type air distributor. Thermocouple ports TR 4-3, 4-4, 3-14, and 4-5 are located in the west wall of the retort 6 1/4-inches above the air inlet level. These ports are used because of their proximity to the four western bayonets. A seven inch immersion places the probe at the same distance from the wall as the bayonets. TI-10 is located at the level of an inlet and is directed toward a bayonet.

Comparing the gas analysis results with the thief sample results show that oxygen concentrations of 0.4 to 1.7% occur when the Fischer Assay range from 0.4 to 7 gal/ton RS.

f. Organic Carbon Balance

The organic carbon balance has continued to be difficult to maintain near the 100% level. During the study of this problem on Retort No. 1 the reliability of shale sampling and analysis was established. It was concluded at that time that gas analysis and gas flow measurement were the primary problem areas.

This is still an area of concern on Retort No. 2; however, other problems have also been encountered.

(1) Effect of Line Out

Much of the data collected on Retort No. 2 has been taken during runs which followed shortly after either a cold startup or a process change. Recently, we have had the opportunity to hold a process condition for a protracted period and observe the effect line out time had on organic carbon distribution. In Table 13 a tabulation of organic carbon distribution is presented for a group of successive runs at the same process conditions. It may be noted that there is a general increasing trend in the organic carbon content of gas and spent shale in each of these series of runs. This is consistent with the slow accumulation of some organic carbon rich species within the retort followed by release of organic carbon in the product streams leaving the retort after an equilibrium level is reached. Such a species might be the dust which has been observed in the combustion and retorting zones of an operating retort. This "equilibration" must be achieved before the unit is lined out.

TABLE 12

TYPICAL COMBUSTION ZONE GAS ANALYSES - RETORT NO. 2

Location		Immersion Inches	CO_2	O_2	N_2	C_1
			Volume %			
Run 702						
6 1/2" Above Air Inlet	(TR 4-3)	7	24	0.4	66	1.3
	(TR 4-4)	7	23	0.4	68	1.4
	(TR 3-14)	7	23	0.33	64	1.4
	(TR 4-5)	7	22	0.35	65	2.3
Directly into a	TI-10	3	23.5	0.39	67	1.6
Bayonet at Air	TI-10	4	21.5	0.4	68	1.9
Inlet Level	TI-10	5	21.0	0.35	72	1.3
Run 685						
6 1/4" Above Air Inlet	(TR 4-3)	7	21	1.4	70	0.9
	(TR 4-4)	7	24	1.3	68	0.3
	(TR 3-14)	7	22	1.7	69	0.4
	(TR 4-5)	7	22	--	72	0.4
Directly into a	TI-10	3	18	4.5	68	1.0
Bayonet at Air	TI-10	4	18	4.6	68	0.85
Air Level	TI-10	5	17	6.3	68	0.70

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TABLE 13

ORGANIC CARBON BALANCE

Organic Carbon Distribution, % Wt

Start Up With Cold Shale

RUN	B672	B673	B674	B675
IN				
Raw Shale	100	100	100	100
OUT				
Liquid Product	61.0	60.8	62.2	63.2
Vent Gas	19.1	17.5	21.7	21.8
Spent Shale	13.5	13.8	13.6	14.4
LOSS	6.4	7.9	2.5	0.6

Start Up With Cold Shale

RUN	B676	B677	B678	B679
IN				
Raw Shale	100	100	100	100
OUT				
Liquid Product	63.8	64.5	64.8	64.9
Vent Gas	17.1	14.9	21.9	19.4
Spent Shale	11.6	12.7	13.0	13.5
LOSS	7.5	7.9	0.3	2.2

Adjusted Bed Height to 7 Feet (No Other Change)

RUN	B680	B681
IN		
Raw Shale	100	100
OUT		
Liquid Product	64.2	63.2
Vent Gas	19.1	20.0
Spent Shale	12.0	13.1
LOSS	4.7	3.7

A correlation between carbon in the gas and organic carbon balance is shown in Figure 1. Although this plot suggests a correlation with a slight slope relating these two variables, the slope of this line is too low to imply that the increase in organic carbon balance is due to an increase in total carbon in the vent gas only. Apparently, something which varies with vent gas carbon is acting with vent gas carbon to improve the organic carbon balance. Spent shale organic carbon appears to increase with vent gas organic carbon as seen in Table 13, therefore this apparent correlation is consistent with the slow equilibration of the unit. As a first attempt at removing this line out effect from data obtained with Retort No. 2 it has been agreed that the unit will not be put on test until twenty hours after a cold startup or sixteen hours after a process change whichever is the longer.

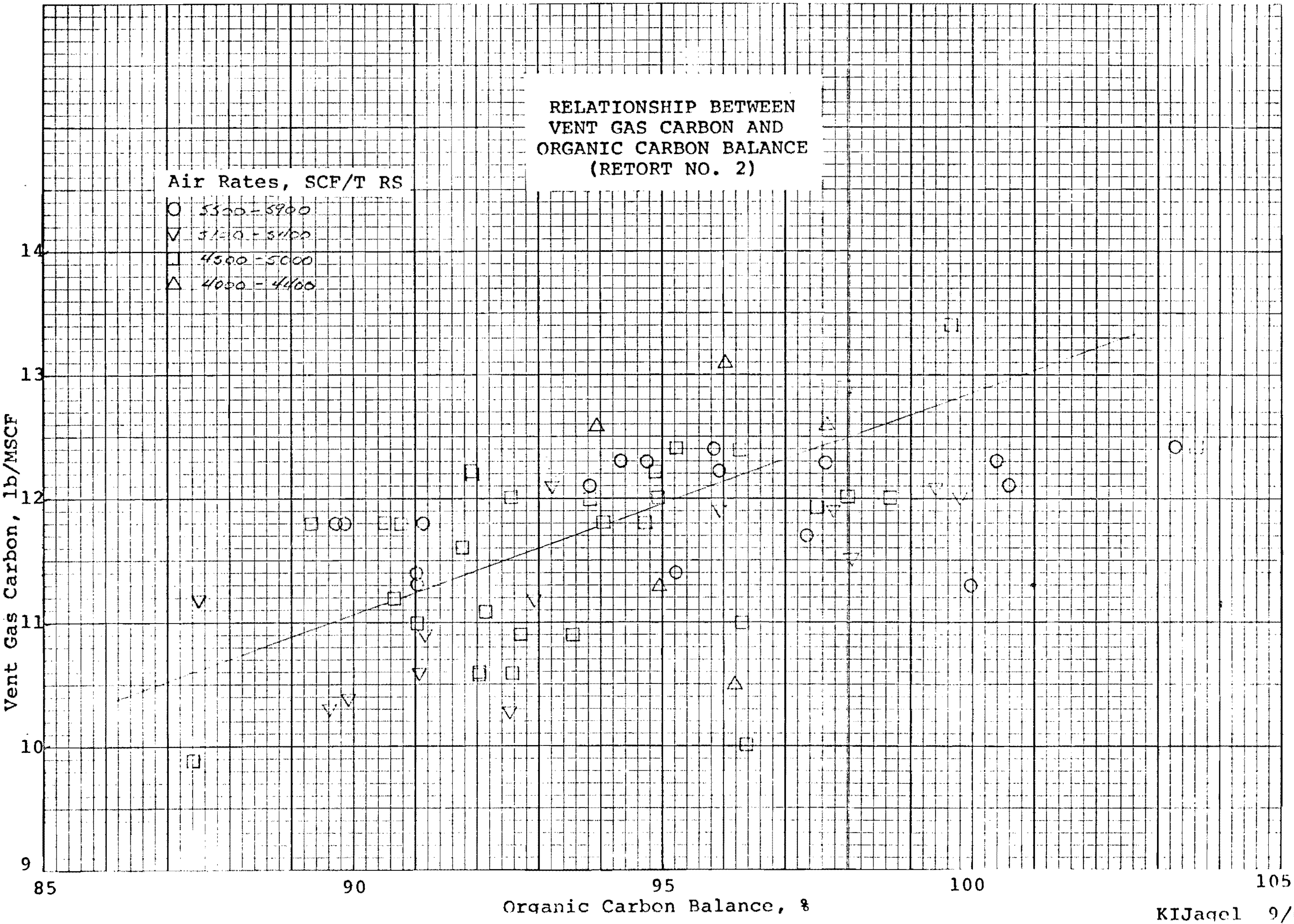
(2) Effect of Organic Carbon Content of Dust Discharged From Rotoclone

Part of the discrepancy in organic carbon balance in Retort No. 2 could be due to an erroneous assumption that the organic carbon content of the spent shale dust discharged from the rotoclone is the same as that on the spent shale. Heretofore, a 100% ash balance was assumed and the organic carbon calculated from carbon analyses of the spent shale. In order to check the accuracy of this procedure, dust samples were obtained from the rotoclone during Run B701. Calculations based on analyses of the dust samples show an average organic carbon content of 3.5% compared with 2.7% in the spent shale. The ash content of the dust averaged 83.7% and an actual ash balance for the run showed a loss of 1,440 pounds. Assuming the ash discrepancy was due entirely to dust loss, the calculated dust loss is 1,720 pounds. This is equivalent to 60 pounds organic carbon. The organic carbon balance calculated on an "as is" basis, therefore, is 95.7%, compared with 95.3% based on raw shale and assuming 100% ash balance.

(3) Water - Oil Emulsions

Obtaining representative samples for the determination of water and sediment has continued to be a problem of major concern both in calculating organic carbon balances and yields. This problem is magnified as the percent water in an oil-water emulsion exceeds approximately 10%. There have been recent runs on Retort No. 2 where water percentages reached 50% in product tank T-3.

'GURE 1



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Under present sampling procedures liquid product samples are caught manually by retort operating personnel. Samples are taken from a vertical discharge line off the liquid transfer pumps. Liquid product tanks are thoroughly pre-mixed before liquid transfer begins.

A test was run recently to reevaluate sampling techniques on liquid product tank T-3. Seven liquid samples were taken as seven 100 pound increments of product were transferred from the product tank. Collection of these samples was closely supervised by engineering personnel. The samples were analyzed in the lab using standard laboratory procedures for percent water. Percent water in the samples as determined by standard procedure were as follows.

Sample No.	1	28.2%	Sample No.	5	26.9%
	2	23.7%		6	22.8%
	3	26.2%		7	24.1%
	4	30.1%			

As shown, water percentages varied between 30.1% and 22.8%. This indicates a substantial difference in water percentage between samples. The following three things will cause differences in water percentages: (1) inadequate mixing in the tanks prior to sampling, (2) human element involved in manually catching representative samples, and (3) the ability to break water-oil emulsions in the lab in order to arrive at representative water percentages.

It is believed the major difference in water percentages between samples is due to our inability to break emulsions. This has been substantiated to a large degree. The seven aforementioned samples were given additional laboratory treatment utilizing heat and chemical demulsifiers. Results of efforts to treat out additional water are listed below:

		Percent Water Following Normal Laboratory Procedure	Percent Water After Additional Heat & Chemical Treating
Sample No.	1	28.2%	31.9%
	2	23.7%	30.8%
	3	26.2%	31.1%
	4	30.1%	30.1%
	5	26.9%	31.8%
	6	22.8%	29.4%
	7	24.1%	27.1%

Note that the spread or difference in water percentages was reduced as a result of additional heating and chemical treatment. This suggests that our manual sampling techniques may not be as bad as first thought if procedures are closely followed. It should also be noted that all samples were not given the same type of chemical treatment. This partially explains why relative increases in water percentages differed from sample to sample.

Visual observation of the oil samples in sunlight indicated the presence of water remaining in the oil after chemical treatment.

Work accomplished thus far suggests the need for improving liquid sampling procedures along with improving procedures for evaluating percent water and sediment in the lab.

Two mechanical liquid samplers have been ordered. These will be installed on product tank systems D-1 and T-3. These samplers are designed to obtain representative liquid samples of fluid flowing in a vertical pipe. A turbulence inducing device will be installed immediately upstream of the samplers. This will insure a thoroughly mixed stream prior to sampling.

Tretolite Company, a firm specializing in chemically treating oil-field emulsions, was contacted by project personnel in regard to emulsion problems. A Tretolite field service man spent one day here at the project attempting to break some of the emulsions with various chemical compounds. He experienced little success.

Tretolite Company requested by letter that a twenty gallon sample of oil be sent to their St. Louis laboratories. Twenty gallons of shale oil have been shipped. This oil will be analyzed in an effort to find a chemical that is effective in breaking water in oil emulsions. An early reply from Tretolite is expected.

B. Mechanical Models Group T. C. Lyons and L. J. Skowronek

1. Shale Segregation Studies

The objective of this work is to determine the degree of segregation that may be encountered in retort and hopper design and to study methods of minimizing it. The major portion of the work has been completed and the results are summarized in the following discussion. Descriptions of the model and procedures used in this study are included.

The model used in this work is illustrated in Figure 2. Oil shale is fed to one side of the model through a feed leg and is allowed to flow to the other side at its normal angle of flow. The far wall is adjustable in order to vary the horizontal distance the particles can travel. The movable tray at the bottom of the model draws the bed down uniformly across the entire width. The tray is compartmented so that 6-inch vertical cores can be sampled across the bed from the feed point to the far wall. A screen analysis of each core results in a particle size profile across the bed.

When a solid is allowed to form an unconfined pile as in this model, a certain amount of size segregation takes place. The amount of segregation is a function of the size range of the flowing particles, the particle shape, and the length of path along which the solid flows (size of pile).

A typical particle size profile obtained with the 1/4 to 3-inch shale is shown in Figure 3. The four individual runs which are plotted in this figure indicate that the reproducibility is reasonably good. The procedure used in this case was to circulate the entire inventory at least once to eliminate the segregation that resulted from filling the model. This insures that the distribution of particle sizes at the bottom of the model is the result of the segregation that took place at the top. After the circulation, two sets of samples were taken (Runs 1 and 2). The inventory was then circulated once again and two more sets of samples were taken (Runs 3 and 4).

In this work, the average particle diameter (APD) is used to compare one core sample to another. The APD is the standard calculation used in the retort work except that the pan fraction has been eliminated and the 1 or 2% of the material which shows up as pan has been normalized over the entire screen analysis. This tends to eliminate the wild scatter in the APD due to the calculation procedure.

In addition to using the APD to compare the individual core samples, it is necessary to have a means of comparing one entire profile against another. To do this, we have instituted a term called EXTENT OF SEGREGATION which is the difference between the APD of the coarsest and finest streams in the bed divided by the APD of the composite. In other words, it is:

$$\frac{APD_{max} - APD_{min}}{APD_{avg}} = \text{EXTENT OF SEGREGATION}$$

FIGURE 2

MODEL FOR STUDYING PARTICLE-SIZE SEGREGATION OF OIL SHALE

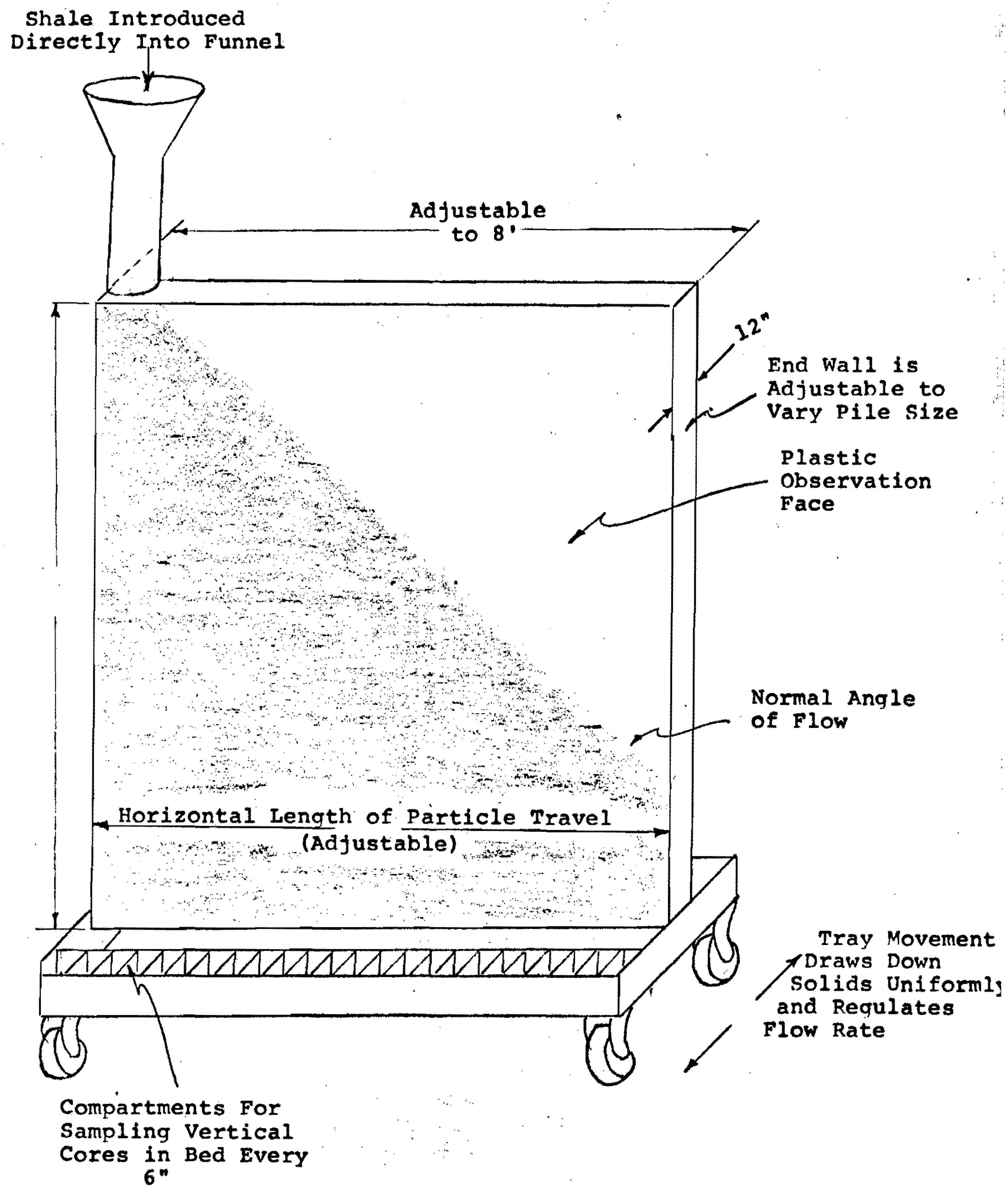

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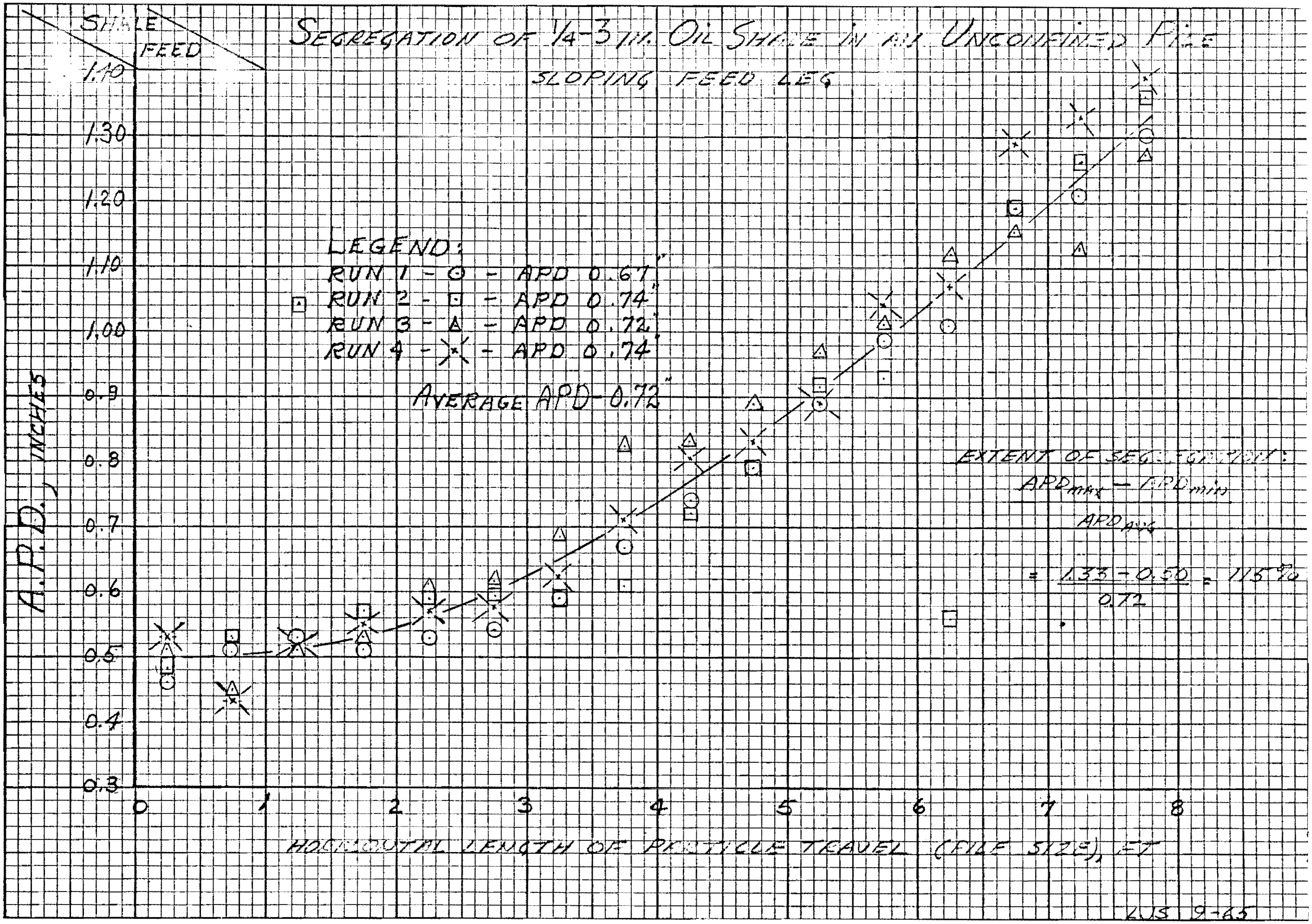
FIGURE 3


SEGREGATION OF 1/4-3 IN. OIL SHALE IN AN UNCONFINED PILE
SLOPING FEED LEG
SHALE FEED
LEGEND:
RUN 1 - ⊙ - APD 0.67"
RUN 2 - ⊡ - APD 0.74"
RUN 3 - Δ - APD 0.72"
RUN 4 - ✳ - APD 0.74"
AVERAGE APD - 0.72"
EXTENT OF SEGREGATION:
$\frac{APD_{max} - APD_{min}}{APD_{AVG}}$
$= \frac{1.33 - 0.50}{0.72} = 115\%$
A.P.D., INCHES
1.40
1.30
1.20
1.10
1.00
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0
1
2
3
4
5
6
7
8
HORIZONTAL LENGTH OF PARTICLE TRAVEL (PILE SIZE), FT
LJS 9-65

In the example in Figure 3, the extent of segregation of the average profile is shown to be 115%. Actually, throughout this work, the extent of segregation was calculated for each individual run rather than the average.

Initially, the effort was concentrated on the 1/4 to 3-inch shale since the tendency to segregate would be greatest with the widest range in particle size. The size profiles that resulted from decreasing the distance that the shale had to travel to reach the wall are shown in Figure 4. The spread of particle size across the bed is quite large even when the travel is as short as two feet. In this entire set of runs, the shale was fed to the model through a feed leg which was sloped at a rather shallow angle of 32° with the horizontal. Thus, it was suspected that segregation was occurring in the feed leg as well as in the model.

In order to minimize segregation in the feed leg, the model was modified so that the feed leg was vertical. The resulting size profiles across the bed are shown in Figure 5. These profiles reveal the characteristic U-shaped segregation pattern that was anticipated. Furthermore, the spread of particle size across the bed has been reduced. This effect is more obvious in Figure 6 where the extent of segregation for the sloped feed leg is compared with the vertical leg. These data show a substantial reduction in segregation in the smaller pile sizes; however, at a travel of six feet the effect is rather small. This comparison illustrates that the geometry of the hardware which feeds a vessel cannot be neglected when considering segregation.

Up to this point, the discussion has dealt with the segregation resulting with the wide range, 1/4 to 3-inch shale. However, there is also considerable interest in the segregation that occurs when operating with a narrow size range. Therefore, similar studies were carried out with 3/4 to 1 1/2-inch shale. The particle size profiles for this material are shown in Figure 7. The extent of segregation is compared with the wider size range in Figure 8. These curves reveal that the segregation with the narrow range is about half that with the wider range.

After establishing the degree of segregation that might be encountered when handling oil shale, the investigation turned to methods of reducing it. Of course, one method is obvious from the preceding discussion; that is, keeping the pile size small. Unfortunately, this appears to be the most effective means of reducing segregation as conventional anti-segregation schemes have not been particularly promising. Confinement of the pile surface has been used effectively to reduce particle segregation of catalyst in

GURE 4

SEGREGATION OF 1/4 TO 3-INCH SHALE IN AN UNCONFINED PILE

Effect of Pile Size

Sloping Feed Leg

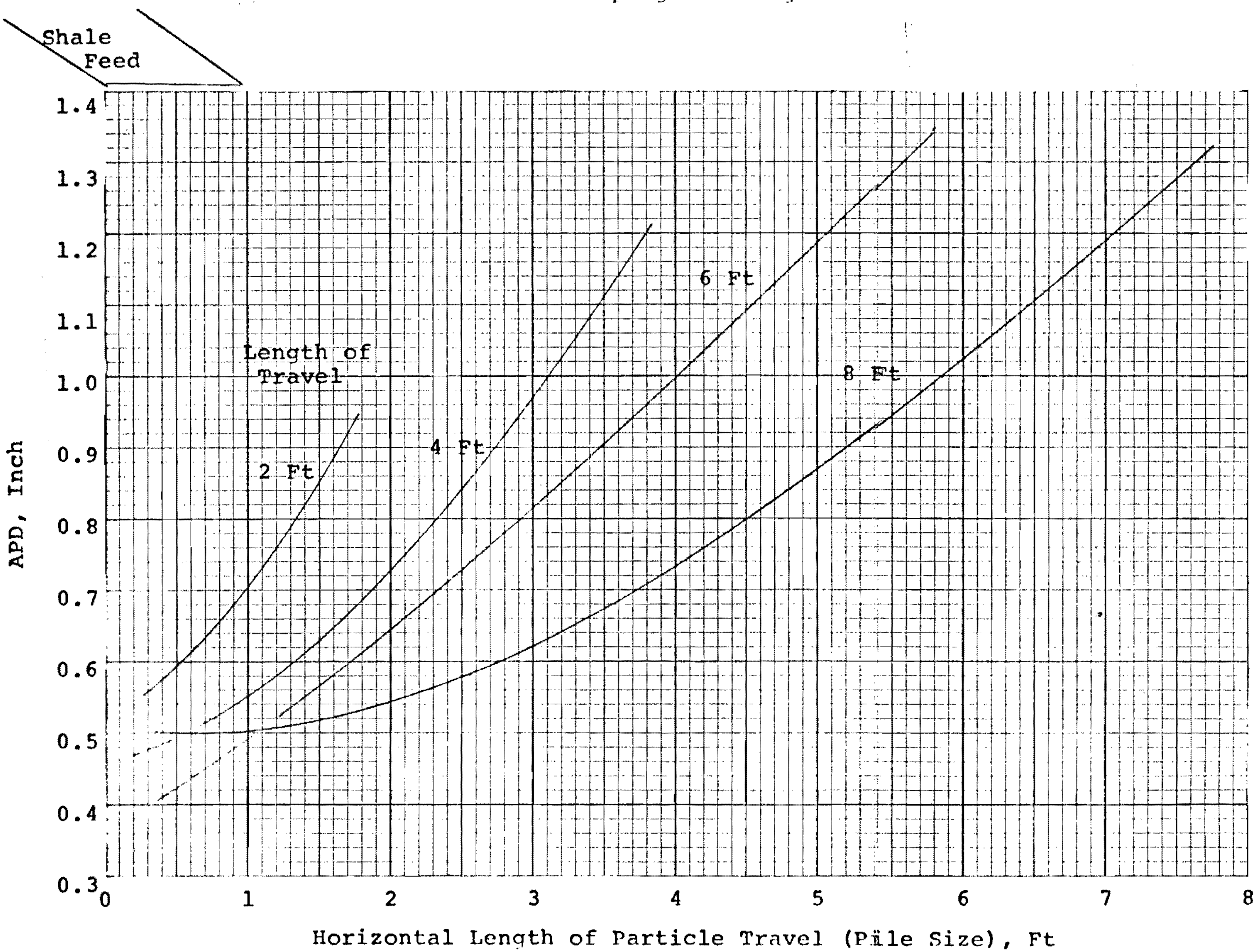


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FIGURE 5

SEGREGATION OF 1/4 TO 3-INCH OIL SHALE IN AN UNCONFINED PILE

Effect of Pile Size
Vertical Feed Leg

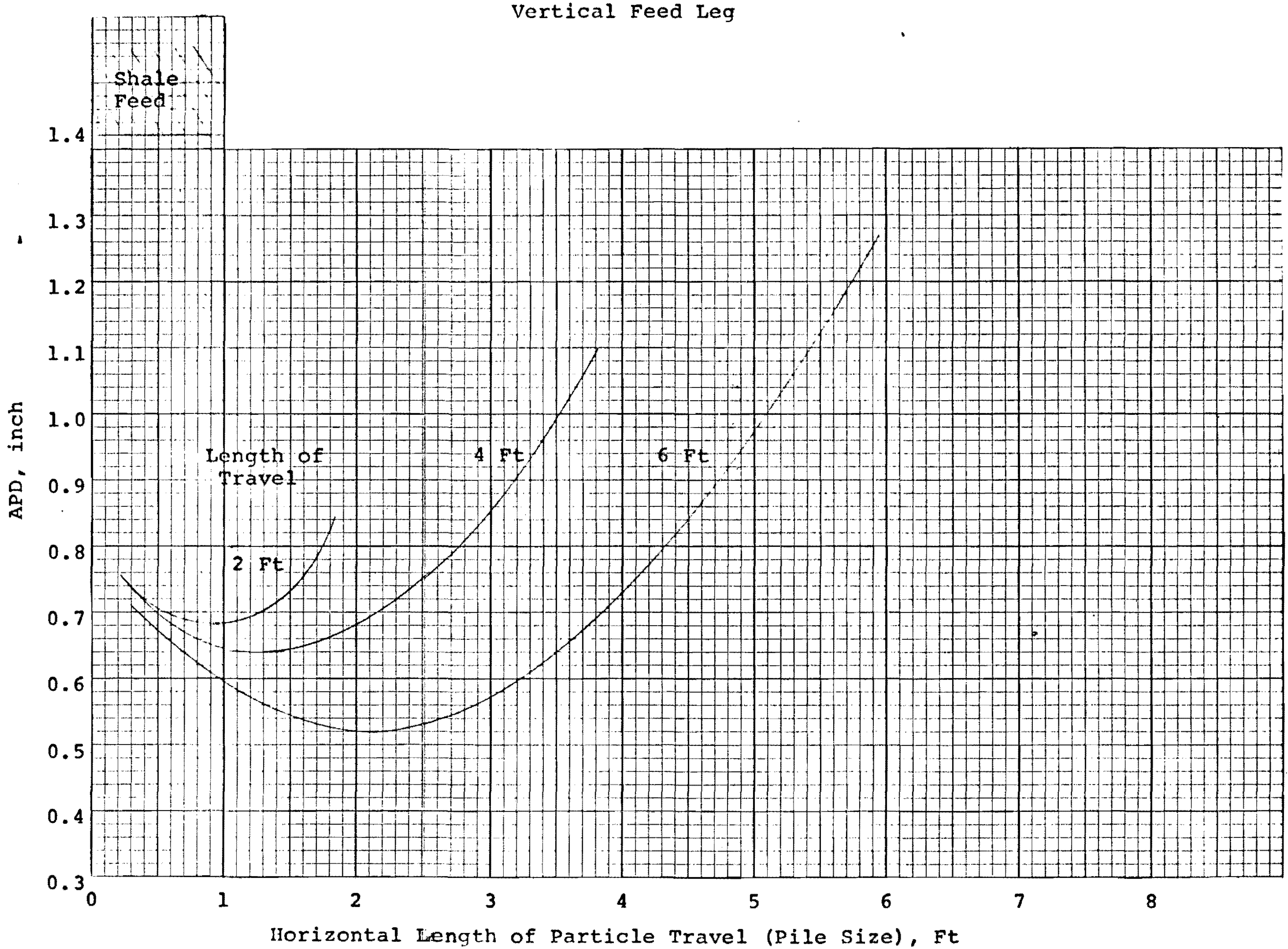

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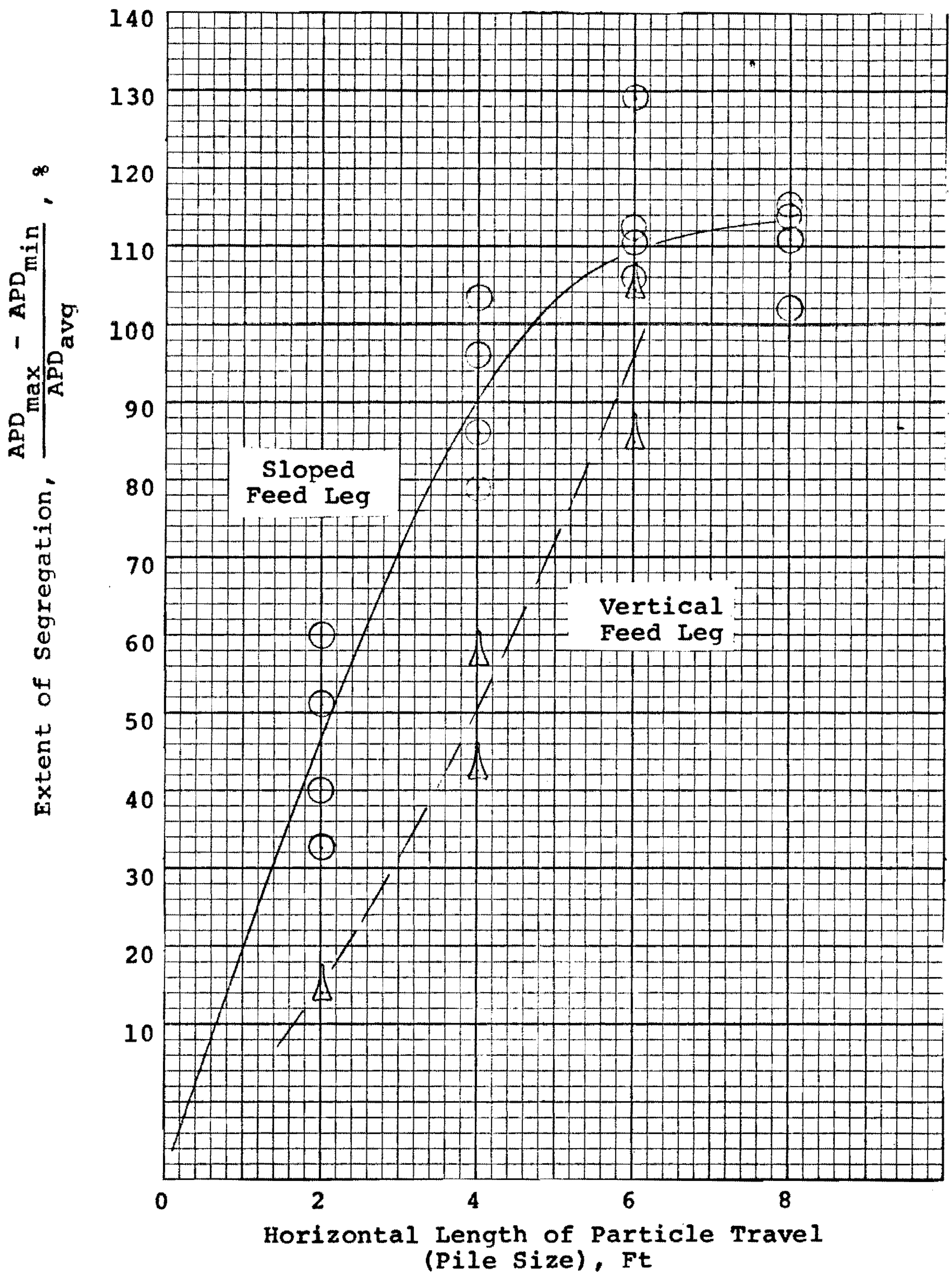
FIGURE 6

SEGREGATION OF 1/4 TO 3-INCH OIL SHALE IN AN UNCONFINED PILE

Effect of Inlet Geometry

Note: Sloped leg was 3 1/2 feet long and 32° with horizontal



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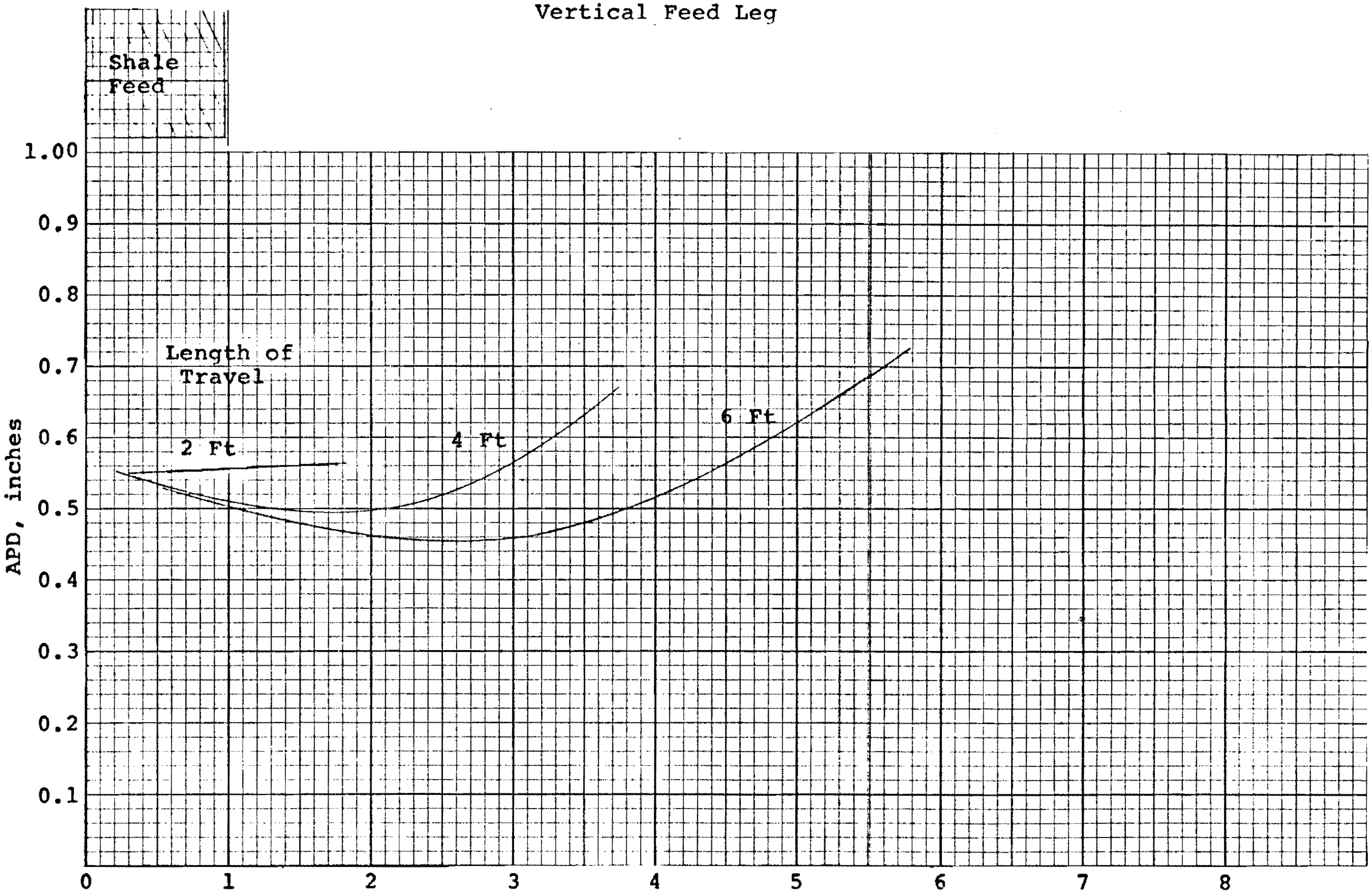
SEGREGATION OF 3/4 TO 1 1/2-INCH OIL SHALE IN AN UNCONFINED PILE

Effect of Pile Size

Vertical Feed Leg



Horizontal Length of Particle Travel (Pile Size), Ft

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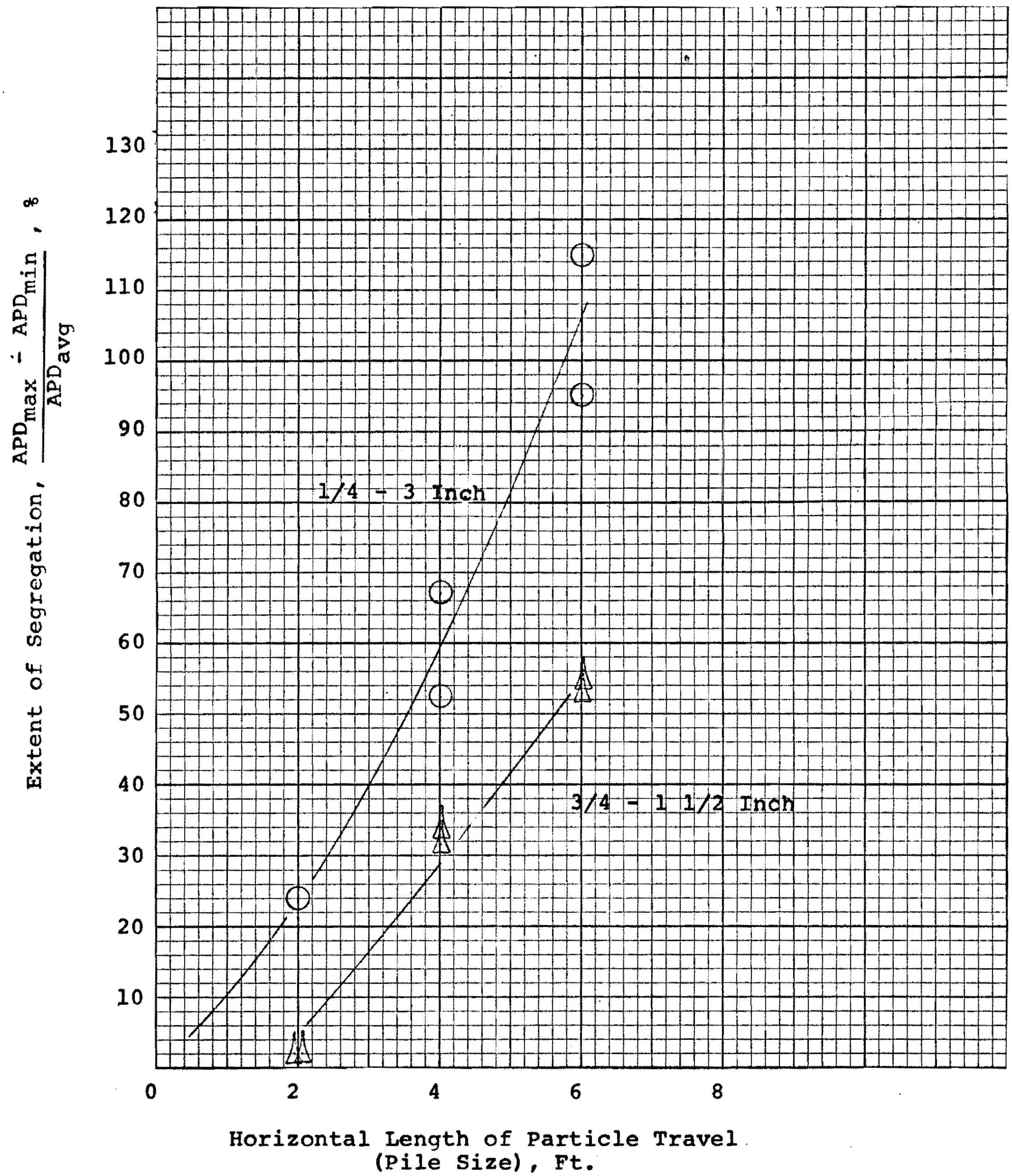
FIGURE 8

SEGREGATION OF OIL SHALE IN AN UNCONFINED PILE

Effect of Particle Size Range

Vertical Feed Leg



TCLyons
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moving-bed cracking units. However, with oil shale, surface confinement has not lived up to expectation. This is illustrated in Figure 9 where the segregation in an unconfined pile of 1/4 to 3-inch shale is compared to that of a confined pile. In this study, cones of 50° and 60° (with the horizontal) were used to confine the surface by 11° and 21° below the normal angle of flow (39°). These data indicate that the 50° cone increased the segregation although there is no reason why this should be so. Because of this lack of improvement, the 60° cone was used solely in the larger pile sizes. With the 60° cone, the effect was not significant until the particles traveled six feet and even then the reduction was not large. The confinement simply made a six foot pile act like an unconfined five foot pile.

Confining the pile surface seemed to be somewhat more effective with the 3/4 to 1 1/2-inch shale (Figure 10) although the improvement was not noted until the length of travel was six feet as in the previous case. However, with six feet of travel, a 60° cone cuts the segregation in half or looking at it another way, it makes a six foot pile act like an unconfined four foot pile.

With the bulk of the segregation work completed it is unlikely that any of the conclusions will change. Limiting pile size seems to be the most effective means of minimizing segregation and vertical or steep feed legs should be chosen rather than shallow legs wherever possible. Studies are continuing to extend the present curves with the vertical leg through eight feet of travel and confined and unconfined piles. Investigation of other surface baffle schemes will be continued although spot runs have not been encouraging to date.

2. <u>Shale Flow Studies</u>

The large demonstration flow model is currently under construction and should be in operation by the end of September. It will be recalled that this model is a continuous flow unit which is 10 feet wide, 20 feet high and 2 feet deep. The primary objective of the studies in this model will be to develop a suitable shale drawoff system for Retort No. 3. However, the model dimensions are such that it can be considered a repeating section for a commercial retort.

Both the mass-flow bin concept and the conventional multiple coning techniques will be evaluated. Small scale flow studies will be made to supplement the large model work. It is in the small model area that the advice of Dr. Jenike holds the most immediate promise. If scalable data can be obtained from the small models with 1/16 to 1/8-inch shale, the flow patterns of many different hopper and

FIGURE 9

EFFECT OF CONFINING CONE ON SEGREGATION

1/4 to 3-inch shale

Vertical Feed Leg

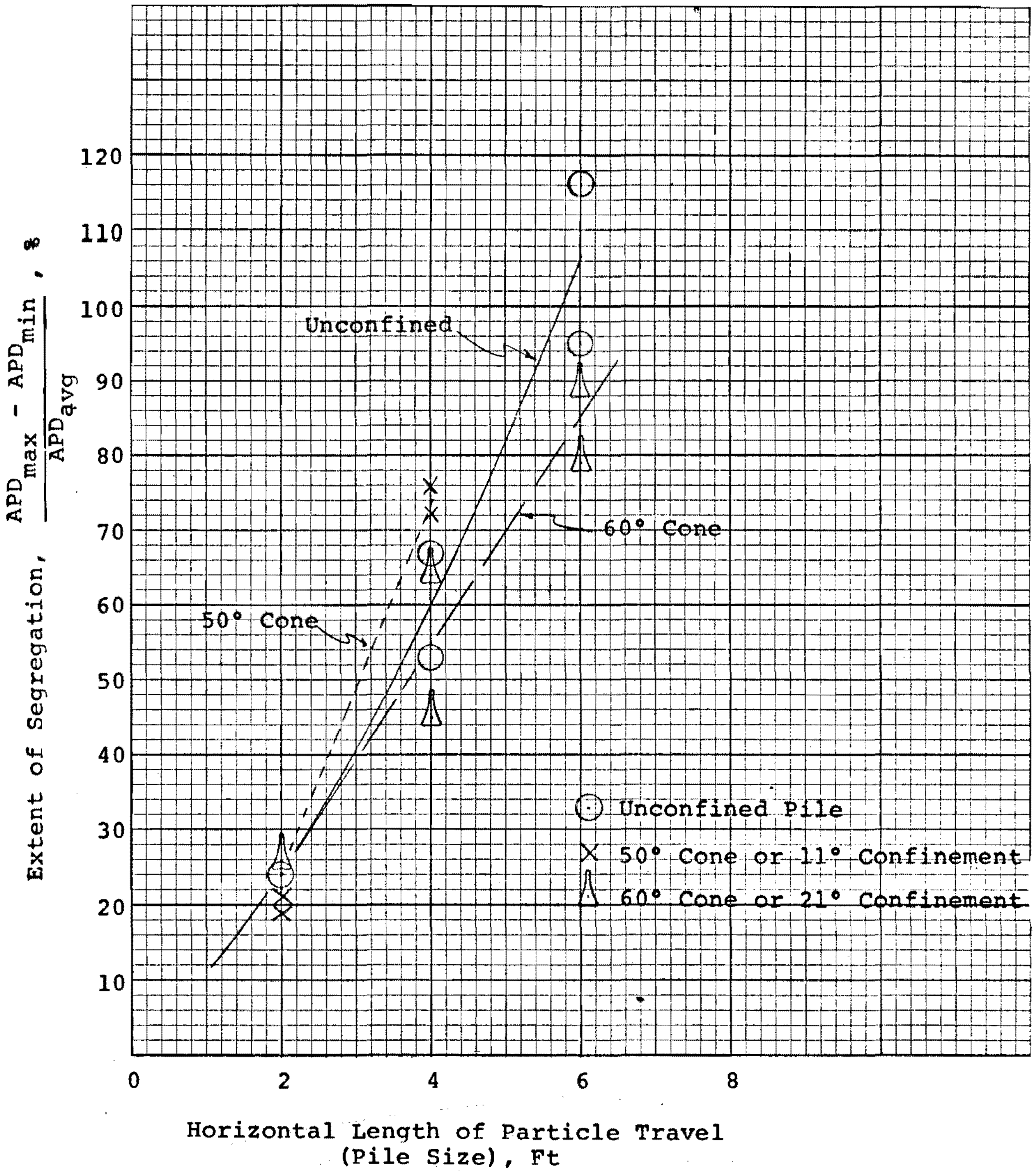

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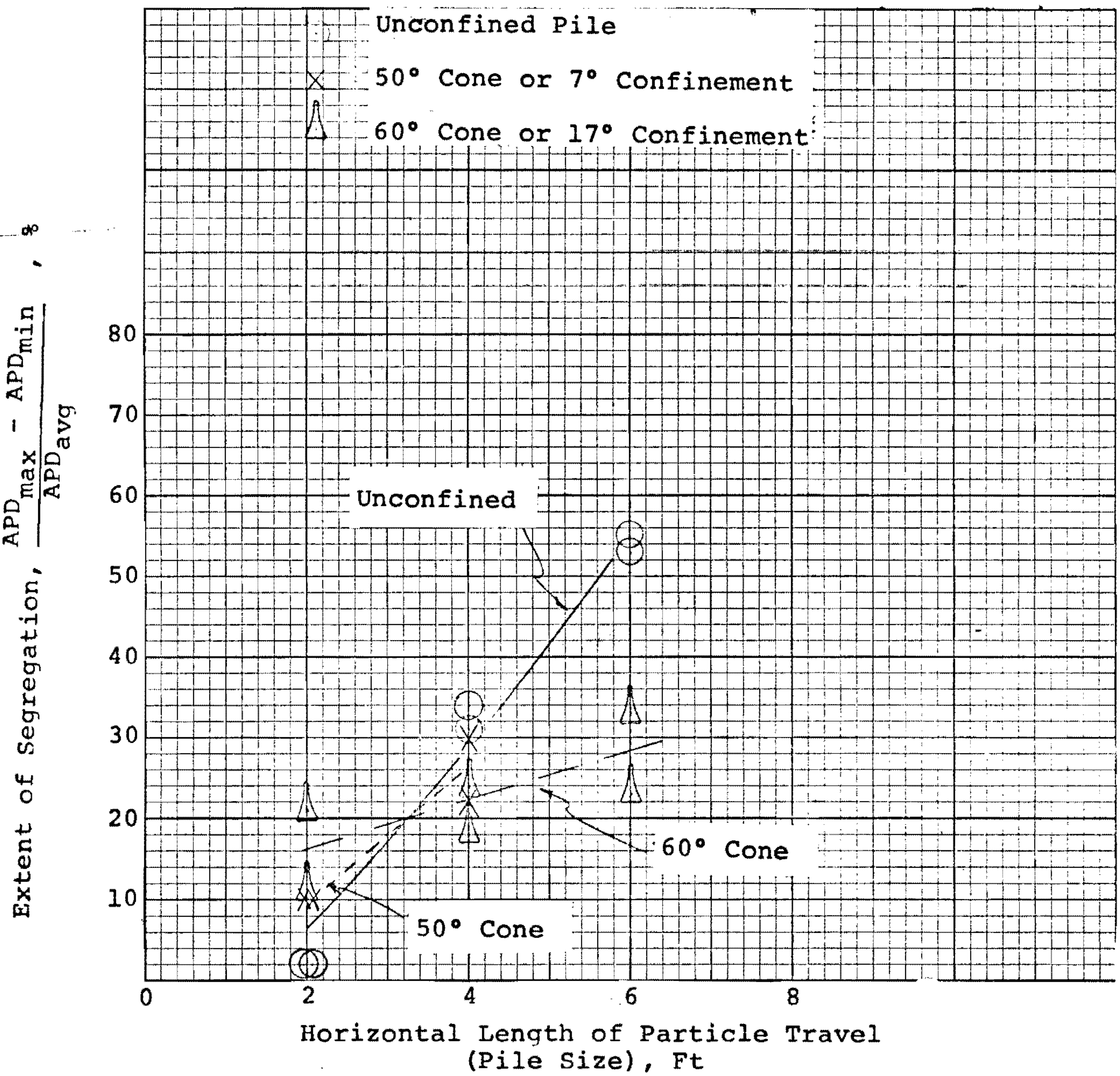
FIGURE 10

EFFECT OF CONFINING CONE ON SEGREGATION

3/4 to 1 1/2-inch shale

Vertical Feed Leg



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retort configurations can be developed quickly with minimum cost. In order to develop the feasibility of the small-model approach, we have submitted samples of 1/16 to 1/8 and 1/4 to 3/4-inch raw shale and spent shale to Dr. Jenike for flowability analysis in his shear testing equipment. We are currently awaiting Dr. Jenike's interpretation of the test results.

3. Dust Demonstration Model

Samples of shale thiefed from the bed of Retort No. 2 have revealed considerable quantities of dust in the retorting and combustion zone. It is hypothesized that this dust has an adverse effect on yields, shale flow, and operability in general. In order to learn something as to the action of dust in a moving shale bed, a demonstration model is being constructed. Initially, the studies will be exploratory in nature. The model will be shaped like an hour glass in order to simulate the velocities in the bottom, top, and combustion zone of a hot retort. It is planned to vary the gas velocity and observe the extent of elutriation from the bed (amount and size of dust). In addition, it is planned to restrict the area in the simulated combustion zone and observe the extent of dust accumulation in the upper section of the retort.

V. ANALYTICAL LABORATORY SECTION (D. Liederman and R. Bernheimer)

A. Work Loads and Overtime

Work loads during the latter two weeks of August and the first week of September necessitated the use of 12-hour shifts for our laboratory technicians. This expedient enabled us to cope with successions of three 8-hour back-to-back runs without an increase in personnel. With the work load returned to normal, we have reverted to 8-hour shifts, which should be adequate for proposed retort operation for the near future.

B. Gas Analysis Standards

While checking the apparent non-linearity of peak height versus concentration for our Matheson gas "standards", we ran two additional points, air (78.0% N_2) and 100% N_2. Because these points did not fall on the curve, we submitted samples of the standard gases to the Paulsboro Laboratory for mass spectrometric analysis.

The results from Paulsboro differed from those certified on the Matheson "standards", but fell much more closely in line with air and pure N_2, as shown on Figure 11. Results for these and other gases are shown on Table 14. Apparently, the errors are mainly in the CO and He determinations. Since N_2 is usually determined as the difference between the total of the other components and 100 percent, the error appears in the N_2 figures also.

To further check the true concentrations of these "standards", we are currently assembling our own gas blending apparatus. In the meantime, we are recalculating all gas analysis data on runs of interest based upon our best estimates of the true standard concentrations. Although Matheson was low by 0.4 and 1.4 percent for nitrogen and high by 0.3 and 1.3 percent for carbon monoxide, on standards A and B respectively, the effect of these errors on data interpretation are nominal as shown in Table 15. The only important effect would be on the interpretation of combustion data.

C. "Others"

Continuing the investigation of "Others" in the recycle gas, we found that the inexpensive Chronofrac is not sufficiently stable and sensitive for this determination. Therefore, we intend to pursue this problem by submitting samples of recycle gas, from retort runs at different conditions, for mass spectrometric analysis. If results from these analyses are significantly interesting, we will propose the purchase of a gas chromatograph with flame ionization detection, for this analysis of "Others".

GURE 11

PEAK HEIGHT VE..US N_2 CONCENTRATION

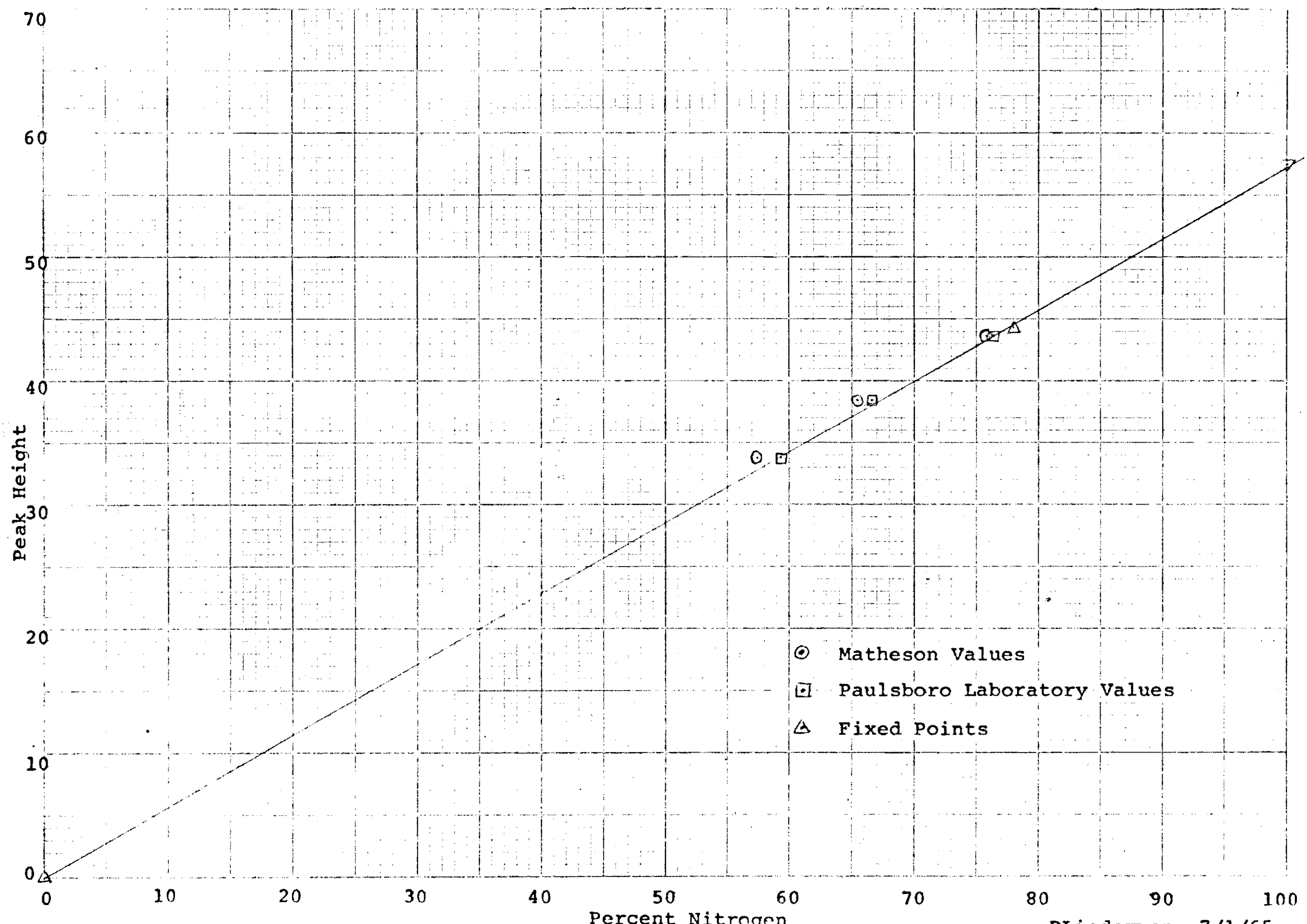

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TABLE 14

ANALYSIS OF MATHESON GASES BY MASS SPECTROMETER
AT PAULSBORO LABORATORY

	Volume Percent					
	Standard A		Standard B		Standard C	
Gas	Matheson	Paulsboro	Matheson	Paulsboro	Matheson	Paulsboro
CO_2	16.3	16.2	25.2	25.1	28.0	28.3
O_2	0.1	0.1	0.7	0.8	1.2	1.1
N_2	75.9	76.6	65.4	66.8	57.4	59.3
CH_4	0.5	0.5	1.9	1.9	1.9	1.7
CO	1.0	0.7	4.9	3.6	0.0	0.0
H_2	5.8	5.9	1.9	2.0	0.0	0.0
Ar	0.0	0.0	0.0	0.0	0.0	0.1
C_2H_6	0.5	0.5	0.0	0.0	0.0	0.0
He	0.0	0.0	0.0	0.0	11.5	9.5

COMPARISON OF SOME RESULTS ON RUNS B547 AND B674 BEFORE AND AFTER RECYCLE GAS CORRECTIONS

Run No.	Before					After				
	FAY	C-Balance	H_2O Balance	Dry Gas SCF/Ton	CO SCF/Ton	FAY	C-Balance	H_2O Balance	Dry Gas SCF/Ton	CO SCF/Ton
B547[a]	90.1	103.9	123.4	8,985	692	90.1	102.8	108.8	8,760	500
B674[b]	81.9	97.5	96.0	6,285	220	81.9	97.5	91.8	6,285	163

[a] CO was decreased by 2.0% and N_2 was increased by 1.5% absolute.

[b] CO was decreased by 1.1% absolute.

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D. Specific Gravity

Because of an apparent discrepancy between specific gravities determined at our laboratory and at Laramie and Golden, we looked again at our computer-prepared tables: We found a slight error in the equation used to generate these tables. The error was extant for Runs 222 through B692, inclusive. It gave results 0.006 low for SG 60F/60F and 1.0 high for API gravity at 60F. It further resulted in reporting Fischer Assay 0.2 gal/ton high at the 28 gal/ton level, but because of compensation in calculation, this caused no change in retort yields. The API gravities are now more in line with those obtained by the Bureau of Mines.

The results of the cooperative analyses between the three laboratories (ours properly corrected) are shown below. The samples are composites of Fischer Assay oils, collected according to gravity over a period of time.

F. A. Oil Sample	Specific Gravity 60F/60F Anvil Points	USBM	CSMRF
1	0.917	0.917	0.916
2	0.914	0.915	0.914
3	0.920	0.921	--

It is apparent that there is excellent agreement among the laboratories.

E. Determination of Water and Oil in Liquid Products

Although we had had problems occasionally in the past in separating water-oil emulsions for the determination of water and oil, they were brought forcefully to the fore again recently. R. L. Clampitt of the Retort Section, who has had extensive experience with emulsions, discovered that the laboratory centrifuge method that we use - and that the Bureau of Mines used - is not accurate. Although, to an untrained eye, there appears to be a clear break between water and oil, in reality there exists more or less emulsion above the water layer. Mr. Clampitt, by extensive emulsion-breaking techniques, was able to break about 14 percent more water out of one high water-content liquid product.

However, at the same time, we did some calibrations on the centrifuge tube and found that the tube read high, practically compensating for the additional water knocked out. In any case, it was shown that sometimes the emulsion was not being completely broken. Even stringent treatment with heat, emulsion breakers, and centrifugation, were not effective for some samples. A representative from Tretolite Company, who spent the better part of a day working with his kit of chemicals, also failed to break the emulsions. Samples of our products have been sent to Tretolite's laboratories for further study.

Meanwhile, we have set up ASTM D-95, a distillation method, to give water results on samples we have tested by the centrifuge method. Although this method takes longer, it is known to give good results.

Although errors may have existed in the determination of water-and-oil, they are not as serious as they may first appear to be. The liquid product distributed about 75 - 25 between the D-3 and T-3 tanks, respectively, contains only about 5 percent water. If we assumed a maximum error of as much as 40 percent for water in the D-3 tank, and 15 percent in the T-3 tank, this would give us an error of only about 1 percent in yield. We believe that the error is usually less than that; but we are determining more accurately what it may be and are studying ways to decrease it further.

VI. ENGINEERING AND ECONOMIC ANALYSES (J. E. Burchfield and P. W. Snyder)

A. Visit to Colorado Fuel and Iron Plant at Pueblo, Colorado

The economic information in the last monthly progress memorandum highlighted the large cost of pumping air, recycle gas and retort gas in a commercial operation. The cost for pumping the gas in a 50,000 B/D plant is possibly 8 - 18¢/Bbl or roughly $2,000,000/year. Because of this high cost and the similarity between retort and blast furnace gas, a visit by J. E. Burchfield was made to the Pueblo Plant of Colorado Fuel and Iron to see what could be learned of the steel industries' experience with blast furnace gas.

The visit was interesting, informative and profitable.

Some items of interest included:

1. A venturi scrubber is used to clean the blast furnace gas prior to burning.

2. Blast furnace gas is burned in blast furnace stoves and steam boilers without unusual difficulty.

3. The gas must be unusually clean or it causes efficiency loss in the boilers.

4. The burners operate with the gas supply at a very low pressure, 8 inches of water psig.

5. Low pressure drop chordal orifices are used to measure gas rate.

The blast furnace gas is considered an extremely important part of the plant's economy.

1. Blast Furnace Operation

There are three blast furnaces almost 23-feet in diameter and approximately 90-feet high. A taper of 1 inch/foot, increasing diameter from the top toward the bottom, assists the flow of expanding solids down to the melting zone. At the contracting melting zone the taper is reversed until the air inlet is approached. The wall then became vertical. The 3-foot thick refractory wall has four radioactive sources inbedded at various depths on three separate levels - a total of twelve. When a given source can no longer be detected erosion of the refractory to that known depth is indicated.

Air is supplied to the blast furnaces via a steam turbine driven air compressor.

Each furnace has an air rate of 60,000 - 70,000 SCF/Min. The air preheated to nearly 2,000° F is mixed with unpreheated air to a blend temperature of 1,350° F. The air is then introduced through peripheral tuyeres into the blast furnace where it burns at a peak temperature of 3,500° F. The oxygen is consumed within about 3-feet of the air inlet.

Skip hoist cars discharge six loads of combined ore, coke and lime, (3/8-inch and larger) into an air lock compartment. See Figure 12. Iron ore smaller than 3/8-inch is sintered to larger size at a sintering plant. The top conical seal seat rotates each dump to distribute the charge evenly on the lower conical seat. The lower seal dumps after six charges and presumably deposits a donut shaped shallow pile on top of the bed.

The gases pass upward through the furnace with CO reducing the oxides of iron to iron which is periodically tapped - about every six hours. Slag is semi-continuously withdrawn as it floats on top of the accumulated molten iron in the bottom of the blast furnace prior to tapping. In tapping, slag floating on the molten iron stream is skimmed off into a slag channel.

The pressure at the air inlet is 20 - 25 psig and about 2 psig at the top. The large pressure drop indicates the high gas mass rates common to blast furnace operation. The literature indicates the mass rate of solids is probably around 700 lb/(hr)(ft^2) and of gas about 1,000 lb/(hr)(ft^2). The high gas velocity and other process conditions apparently contribute to bridging in the blast furnace which periodically break through resulting in large surges of gas and pressure. These "slips" occur frequently perhaps every 10 minutes or so.

The gas produced from the blast furnaces is similar to retort gas in heating value and composition as shown below.

Component	Blast Furnace	Retort Gas
Nitrogen, Vol %	58	60
Oxygen	1	0.2
CO_2	10	26
CO	27	5
H_2	1	5
CH_4	0.5	2
Others		2
Typical Gross Heating Value, Btu/SCF	90 - 105	80 - 120

The gas temperature is normally about 400° as it comes out of the furnace.

2. Blast furnace Gas Clean Up

Blast furnace gas from the top of the bed passes though large ducting into a vertical venturi scrubber for cleanup. The gas contains about 15 grains of solids/SCF gas. The gas was formerly cleaned in an electrostatic precipitator to about 0.5 grain/SCF. However, this level of cleanup was unsatisfactory. The solids, found to be oxides of iron, deposited during burning in the bottom of the boilers. Each year a large accumulation of these deposits had to be cleaned out manually. Success with venturi scrubbers elsewhere lead CFI to install this type of cleanup device about a year ago.

The venturi scrubber is simply a venturi restriction installed vertically in the ducting. Water is injected in multiple ports at the restriction. The water and gas go into large diameter cone bottom separator beneath the scrubber. The gas separates and is taken off the top of the vessel through large ducting. The water seals the gas down leg and is withdrawn from the bottom of the vessel.

The scrubber takes a pressure drop of about 45.0-inches of water to 2 psi. The blast furnace gas pressure is maintained at about 14-inches of H_2O downstream of the scrubber. The gas is cleaned to 0.03 grains/SCF. It contains about 2.1 pound of free water/MSCF as it goes to the consuming unit.

3. Burning Blast Furnace Gas

About one third of the gas is burned in blast furnace stoves used for preheating blast air. The other two thirds along with some natural gas is burned in boilers to produce steam and electricity for the steel works.

4. Blast Furnace Stoves

Each blast furnace has four stoves in which air is preheated. Three stoves are regenerating or storing heat while one is preheating blast air. The air preheat cycle is six hours. Blast furnace gas and natural gas are mixed with air and burned in large burners. The ignited gases pass upward through a burning chamber and then turn downward passing over and heating brickwork called checkers. See Figure 13. The brickwork consists of large bricks perhaps 3 X 6 X 10-inches containing two openings through which gas passes. The brickwork stores heat, up to a temperature approaching 2,000° F. flame temperature. After the stove preheating air cools it is swung into this regenerative cycle while a hot stove is swung over to preheat air.

Air is introduced at the bottom of the checker works and passes upward in a direction opposite to that in which the burned gas traveled. It is preheated initially to nearly 2,000° F and gradually falls off in temperature. By passed air is mixed with preheated air to give a temperature of 1,350° F. This air is then introduced through peripheral tuyeres into the blast furnace. No difficulty is encountered in burning the gas.

When "slips" in the blast furnaces release large surges of blast furnace gas, the system has pressure controllers and releast vents which prevent serious upsets in the consuming units.

5. <u>Boilers</u>

Blast furnace gas is burned in several boilers at the rate of 1.0 - 1.2 MSCF/hr. The older boilers with radiant walls burn blast furnace gas alone. Newer boilers with black furnace wall use a natural gas pilot to guard against a flame out. Since supplemental natural gas is required to meet power need this is not regarded as a disadvantage. These newer boilers are more efficient and are base loaded so that swings in load are taken on the older furnaces.

A chordal orifice taking 0 - 4 inches of water pressure drop is used to measure gas rate in large 36-inch piping. The chord is located in the top of the pipe to minimize the effect of entrained solids and water buildup on measurements. Nevertheless, the meters do lose accuracy as deposits accumulate on the bottom of the pipe.

There are three types of burners used to burn blast furnace but they function in essentially the same manner. The burners used at CFI are Peabody, Freyn and a home-made burner. The burner body is a ring about 18-inches in diameter and 2-inches thick, and perhaps 10 or 12 inches long. The blast furnace gas passes through the center of the cylinder. Small natural gas ports are located around the periphery of the burner. Air preheated to 550° F is introduced into a concentric chamber surrounding the burner gas supply duct. The air and blast furnace ignite in the fire box after mixing at the burner. A refractory lined shield protects the burner from burn out by radiant heat. See Figure 14.

The gas is available downstream of the chordal orifice meter at approximately 8-inches of water pressure. The boiler operates at a negative pressure of about 0.1 - 0.2-inches of water gauge.

The smaller amount of solids and entrained water cause problems in the boiler burner. However the problem is smaller than that due to the solids remaining after cleanup by the electrostatic precipitator. The problem is also different. A hard deposit is formed on the first row of tubes lower boiler efficiency by 3%/year (estimated to cost $60,000/year). The deposits are zinc compounds, which tenaciously adhere

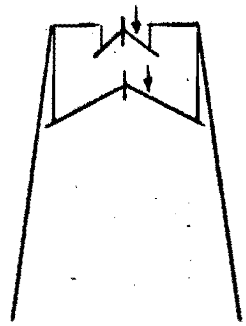

Figure 12

Air Lock Seal for Charging Solids

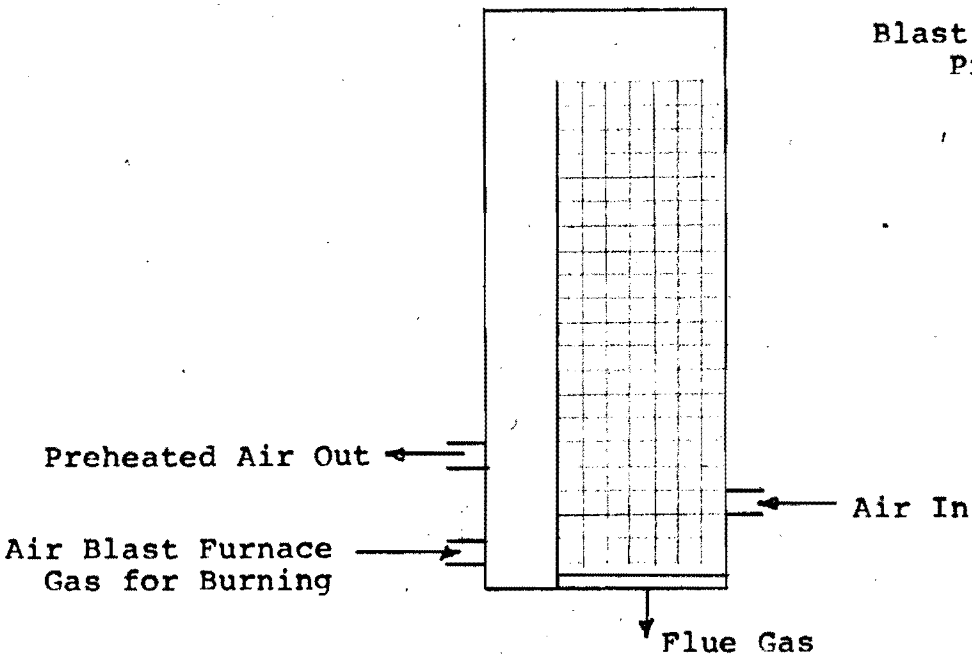

Figure 13

Blast Furnace Stove For Preheating Air

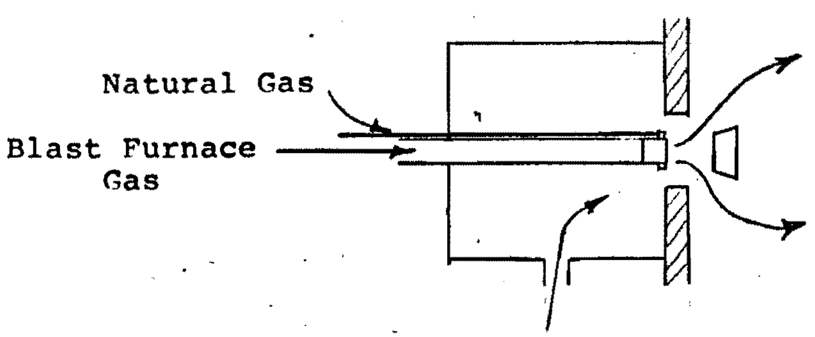

Figure 14

Burner for Burning Blast Furnace Gas

JEBurchfield
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to the tubes. Coke breeze (finely divided coke particles) is used to clean the deposit from the tube during operation. But there is some concern this erosive action will also reduce tube life.

The solids and water are believed to be jointly responsible for the type deposit and the mechanism by which it is layed down. In addition the entrained water must be vaporized in the boiler at a high cost - estimated at $60,000/year.

Design features such as water seal legs guard against infiltration of the toxic gas during shutdown of a boiler. Alarms are also used extensively to guard against hazardous leaks during boiler shutdown.

6. Miscellaneous

CFI uses a hydraulic coupler on the gas blower for the coke oven gas which is also burned in their furnaces. The couple permits them to change pressure output on the blower and thereby save some gas compression costs.

Although CFI does not use gas turbines on blast furnace gas apparently some steel companies do. The blast furnace superintendent, Mr. John Carlson, indicated that turbines were in satisfactory use elsewhere in the industry.

B. Effect of Air Rate on Gas Production

The gas production from Runs 543 to 576 from Retort No. 1 have been compared with the gas production for Runs B620 to B700 from Retort No. 2 in an attempt to seek differences between the two retorts. This comparison showed no differences in the gas production between Retort No. 1 and Retort No. 2. However it does point out the following interesting characteristics of the combustion reactions which take place within the retort:

1. Carbonate decomposition is directly proportional to the air rate in SCF/T at any one recycle gas rate. Increasing recycle rate increases carbonate decomposition. This relationship is not unexpected. Since the heat requirements in the top of the retort does not vary significantly, any incremental heat of combustion must go to either carbon decomposition or increase spent shale temperature. The relationship is the same for Retort No. 1 and No. 2; indicating that air rate measurements are consistent between the two retorts. This relationship is shown in Figure 15.

2. The nature of the net combustion reactions changes with air rate above 4,000 SCF/T. Above 4,000 SCF/T the incremental air is utilized in producing primarily CO_2 and CO and very little in producing water as shown in Figure 16. The change in combustion characteristics above 4,000 SCF/T of air results in a 16% reduction in the heat released per incremental SCF of air. It also indicates the burning of a very hydrogen-rich material with the initial

FIGURE 15
CARBONATE DECOMPOSITION

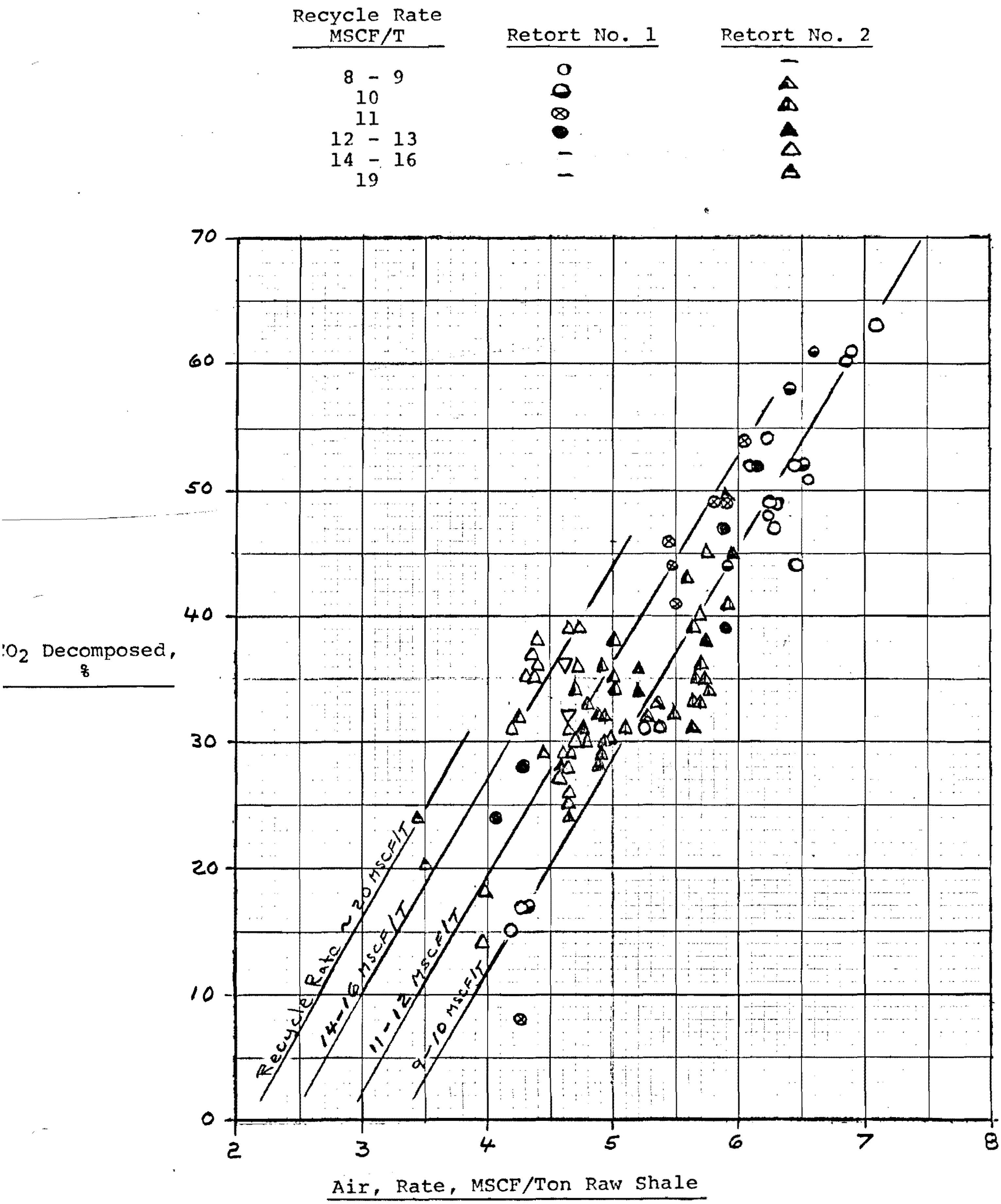

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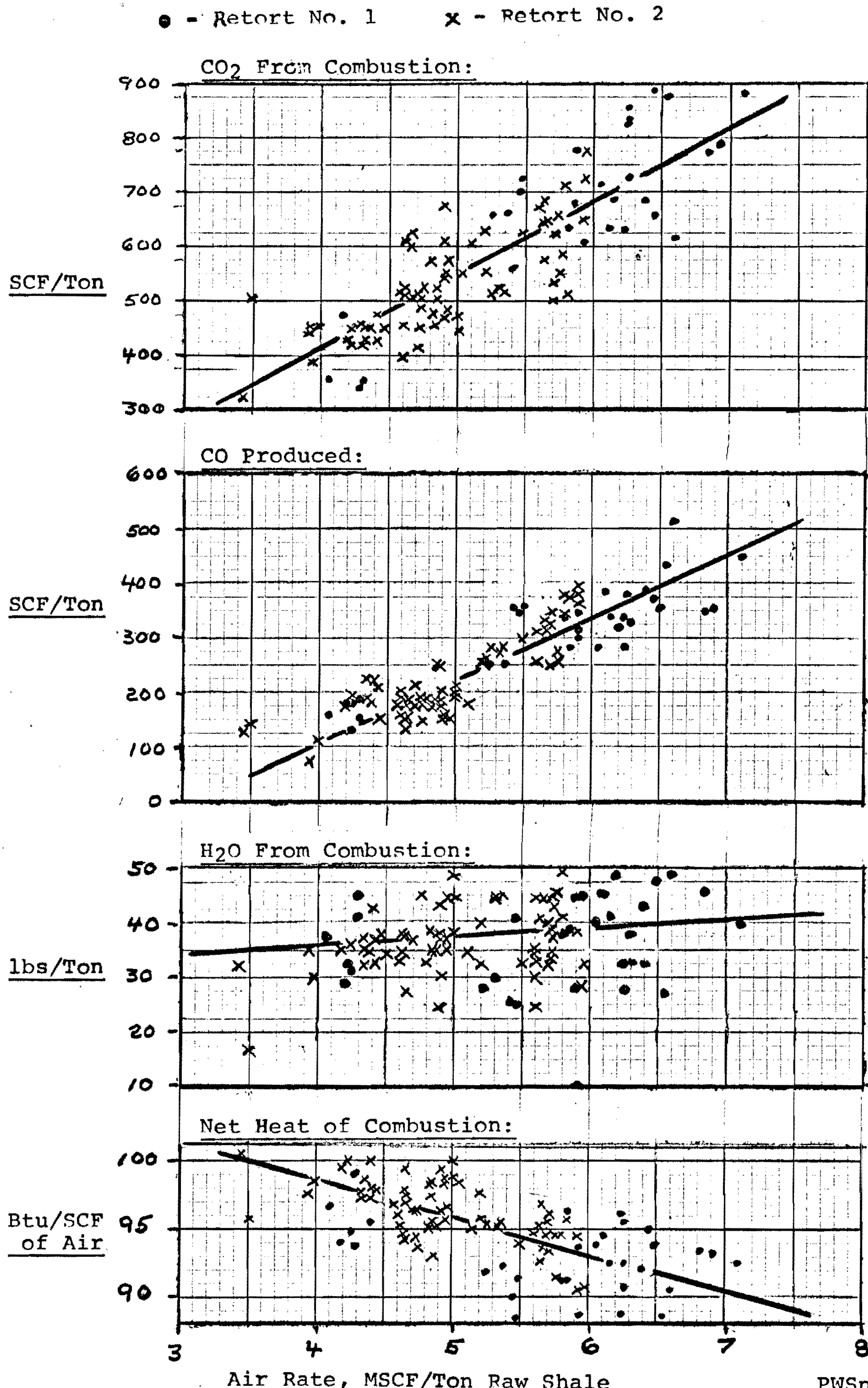
FIGURE 16
PRODUCTS AND HEAT OF COMBUSTION



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oxygen and a hydrogen-poor material with the incremental air above 4,000 SCF/T. The oxygen utilization and its significance is shown below:

Air	Initial 4,000 SCF/T	Incremental Over 4,000 SCF/T
O_2 Utilization, %		
CO_2	49	65
CO	6	27
H_2O	45	8
Total	100	100
Net Heat Released, Btu/SCF of Air	100	84
Net C/H of Material Burned, wt/wt	4	46

The carbon-to-hydrogen ratio for coke on spent shale is about 15 and is 3 for methane. The exceedingly high carbon-to-hydrogen ratio of material burned with incremental air is most likely the result of simultaneous steam reforming reaction converting water to H_2 and CO.

3. The hydrogen production also increases with increased air as shown in Figure 17. This is further evidence of the steam reforming reactions increasing with higher air rates. Adjusting the hydrogen yield for fluctuations in raw shale assay from 24 to 33 gal/ton improved the correlation indicating that the hydrogen production is also a function of assay. This is expected since the amount of hydrogen produced by the Fischer Assay is directly proportional to the assay.

4. The methane production was not a function of air rate. It does appear to be a function of Fischer Assay yield as shown in Figure 17.

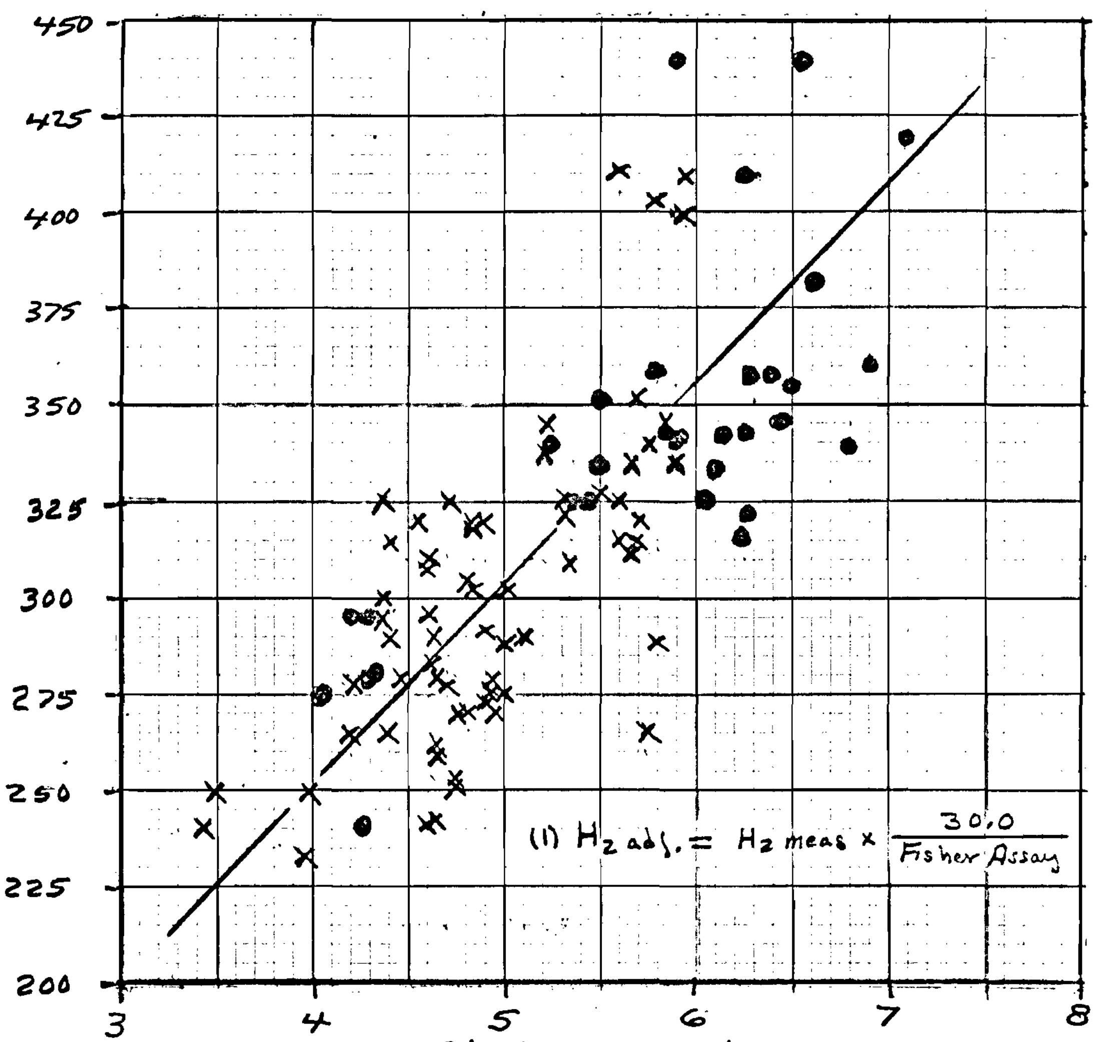
The significance of these observations with respect to retort yield and performance has not been established at this time. Additional evaluation and analyses will be spent toward developing this significance and any conclusion will be reported in a later progress report.

FIGURE 17

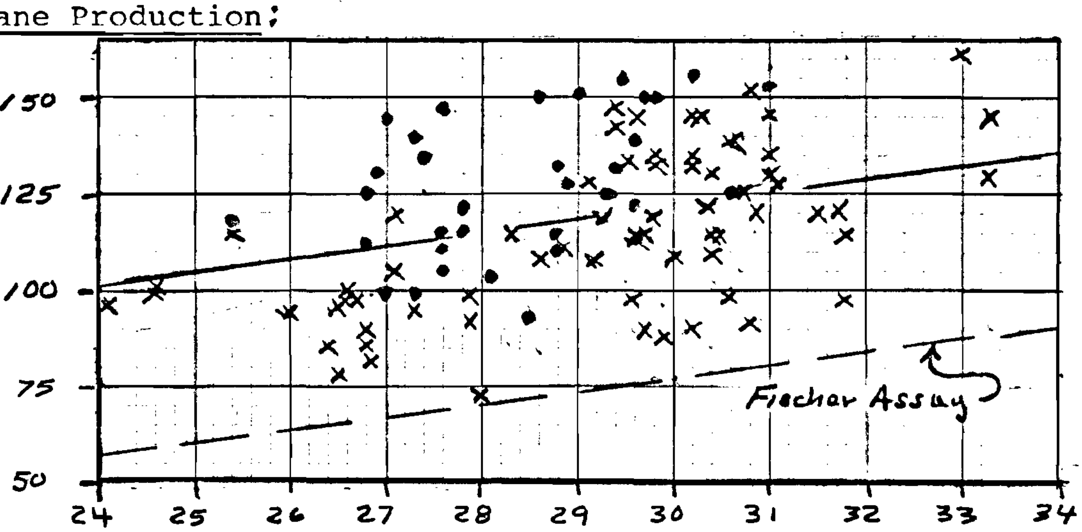
HYDROGEN AND METHANE PRODUCTION

● - Retort No. 1 x - Retort No. 2

A. Hydrogen Production (Adjusted to 30 gal/ton raw shale[1]):



B. Methane Production:



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