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MOBIL OIL CORPORATION
RESEARCH DEPARTMENT

MONTHLY PROGRESS MEMORANDUM

(Covering December 16, 1966 to January 15, 1967)

ANVIL POINTS OIL SHALE RESEARCH CENTER

Rifle, Colorado

January 20, 1967

CONTRIBUTORS:

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

Signed by:

R H Cramer

R. H. Cramer
Program Manager

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] Mobil Oil Corporation

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

MONTHLY PROGRESS MEMORANDUM

(Covering December 16, 1966 to January 15, 1967)

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CONFIDENTIAL

MONTHLY PROGRESS MEMORANDUM
(Covering December 16, 1966 to January 15, 1967)

I. TECHNICAL ADVISORY COMMITTEE

The sixteenth regular meeting of the Technical Advisory Committee was held in Tulsa, Oklahoma on January 12, 1967.

The next meeting is scheduled for March 2 and 3, 1967 at Anvil Points. A tour of the facilities will be arranged for March 3.

II. MINING SECTION (C. R. Haworth, J. B. Sellers and F. W. Brackebusch)

A. Mining Program

During the period starting December 16, 1966, mining had progressed to the point where the entry to Room No. 2 had been widened out to 60 feet, and where the crosscuts through to Rooms No. 1 and No. 3 had been advanced 20 feet and 35 feet respectively. Over the December 24 - 25 weekend a roof fall took place in the area where Adit 5 widens into Room No. 2. This fall caused an interruption in the mining schedule, and corrective action is now being taken to put the area back in a safe condition. The complete details of the fall and its consequences are available in Technical Memorandum 67-2 issued on January 10, 1967.

The rehabilitation work included the job of making safe the 11 foot thick roof beam above the 2 foot slab which had pealed off. To accomplish this, we first brought in four 25 foot long, high strength rock bolts. These bolts were anchored in the back and pull tested to 40 tons. The anchorage was excellent and additional bolts were purchased to complete the work.

Sixteen of the 25 foot rockbolts have been installed around the southeast periphery of the fall area. The sag rods installed in the center of the span show that the rockbolts are taking the weight off the beam. The parting at the 12 foot 9 inch horizon above the back has closed 3/1000 inch since the bolts were tensioned. The 2 foot slab which was hanging free from the back, around the perimeter of the fall area, has been blasted down. It was removed by drilling a row of holes into the slab behind the damaged zone. The holes were 16 inches long and 2 feet apart. Half a stick of 1 inch by 8 inch Gelex No. 2 was used in each hole. Work is now proceeding on the installation of the 25 foot rockbolts throughout the area of concern on an 8 foot spacing. Channel and chicken wire will then be used wherever necessary to prevent any slabbing. The rehabilitation program is on schedule and should be complete by February 7, 1967. There is sufficient shale stockpiled to feed Retort No. 3 on a continuous basis until February 10, 1967.

In the instrument tunnel all of the instrument holes have been drilled up through the center row of pillars. The holes are now being surveyed before installation of the extensometers.

B. Bench Scale Test Work on Physical Properties of Oil Shale

The errors in instrumentation have now all been eliminated and the compressive strength tests are well underway;

Table 1 summarizes the test results received to date. It is apparent from these results that there is a correlation between compressive strength and oil content. The presence of inclusions can have a weakening effect on the samples being tested as can be seen in the result of loading specimen No. 78. In the case of full scale pillars, these inclusions would have a negligible effect on the strength of the shale unless they were very extensive.

The effect of height: width ratios on the compressive strength is not yet apparent from the data. When the tests are complete a detailed analysis will be made.

C. Assay of Mine Run Shale

In view of the persistent low oil content in the retort feed during the past two months, a core sample was taken from one of the instrument holes located in the center of the new mining area. Fischer assays were run on 1 foot sections from 20 feet above to 20 feet below the mahogany marker, this being the horizon we are presently mining. The assays averaged 27.6 GAL/T confirming the results taken from the United States Bureau of Mine's coreholes A and D-5, which ran 27.7 GAL/T and 27.8 GAL/T respectively. The lean retort feed must be a result of either; a very localized anomaly in the oil shale beds, segregation in the stockpiles or dilution during the stockpiling process. Further study will be necessary if the feed continues assaying out at 24 to 26 GAL/T.

TABLE 1

BENCH SCALE TEST WORK ON PHYSICAL PROPERTIES OF OIL SHALE RESULTS RECEIVED TO DATE

Specimen No.	Oil Content	Height: Width Ratio	Compressive Strength	Remarks
3	23 GAL/T	1:1	14,700 psi	$E \sim 1.2 \times 10^6$
6	26 GAL/T	1:1	17,900 psi	$E \sim 1.3 \times 10^6$
23	18 GAL/T	3:1	24,600 psi	
24	16 GAL/T	1.825:1	22,000 psi	
33	17 GAL/T	1.825:1	27,200 psi	
40	56 GAL/T	1.825:1	9,800 psi	
49	28 GAL/T	3:1	11,600 psi	
70	32 GAL/T	3:1	14,200 psi	
78	44 GAL/T	3:1	6,000 psi	The specimen started breaking up along an inclusion.
106	41 GAL/T	1.825:1	10,000 psi	$E \sim 1.2 \times 10^6$

GRHaworth
1/16/67

III. MECHANICAL ENGINEERING (W. S. Bergen, R. E. Smith and J. J. McAleer

Retort No. 3 operated for a 12-day period testing 1 to 2 1/2 inch shale at a 400 shale mass rate. During this period of operation, no major modifications were made to the retort. Repairs and revisions were made to several systems to maintain or improve operability.

The mine grizzly was completed and shale processed for the crushing study program.

A. Major Conclusions

1. Crushing Plant

The crushing plant can produce the quality and quantity of shale for the 1 to 2 1/2 inch operation. Preventative maintenance is necessary for the required sustained operation.

2. Shale Weighing and Accounting

This system does provide accurate data needed for material balance needs.

3. Shale Flow and Control Systems

Both raw and spent shale systems function as specified and form a compatable overall system.

4. Instrumentation

a. Instruments and computer circuits function as specified.

b. The use of thermowells extending through the combustion zone appears impractical.

c. Offgas temperatures are a sensitive indication of process or retort malfunctions.

d. Pressure records at various retort levels are necessary for proper insight of unit operation.

5. Retort Internals

a. The 3-header systems function as designed.

b. The 3-header systems are structurally sound.

c. The air riser manifold will require additional gusseting.

d. Brick work in all but the combustion zone appears satisfactory.

e. Brick in the combustion zone must have few or no indentations or depressions. Wear occurs on brick adjacent to these depressed areas.

6. Recovery System

a. Jet impactor tests show the precipitator to be 99.8% efficient.

b. The overall recovery system, multiclone plus precipitator, is 99.98%+ efficient.

7. Liquid Product System

a. Decanter system will require further tests and development to handle the sludge.

b. LACT system meters properly.

8. Spent Shale System

a. The slusher system does not operate as yet. Revisions being made by Torkelson Company at their expense.

b. Use of drain water appears effective in minimizing but not eliminating dust problems.

c. Wet spent shale has a high compaction potential

9. Blowers

a. Liquid deposition in a multi-stage recycle blower unit will require the addition of stage drains.

B. Discussion

Revisions or repairs have been made to many systems during this period. System reliability and operability are system goals.

1. The recycle blower failed after the December 11-12 shutdown. Upon dismantling the unit, we found an oil water buildup in the various stages and that the inboard and outboard bearings were improperly assembled. The first reassembly of the unit did not produce a workable unit. After discussions

with Spencer, the O'Brien Machinery Company, and the Mobil Paulsboro Laboratory, an effective reassembly was realized. The reassembly of the various staging spacers was necessarily not in accord with Spencer's drawings. Spencer is aware of and in accord with our changes. This shutdown required one week.

2. Extensive changes have been made to the raw shale sampling system to produce effective riffling of the product. The internals from the Retort No. 2 riffler were installed in the Retort No. 3 riffler casing. Tests to date have shown the change to be effective.

3. All instrument impulse and air lines have been heat traced.

4. The lower two thermowells were removed after the unit shutdown of January 12. They are being reinstalled effective January 16. A clinker had bent one thermowell.

5. The line burner casing was seriously damaged January 10. During the test of the burner, prior to startup, the secondary air had been bypassed around the burner. This air cools the casing. Warped end sections which had failed were replaced. The burner was also given additional reinforcing. No permanent damage occurred.

6. The new SCR control element of the Merrick rate control system was installed. This has worked satisfactorily since installation.

7. The gear reducer casing of the top star feeder split in service New Years Eve. The top star feeder was removed and a sleeve installed until the feeder could be replaced January 2. This work was accomplished without shutting the unit down.

8. Extensive tests have been made of the Fischer Porter D/P cells to check and reset their calibration. No problems have occurred since the recalibration. Accurate signal measuring equipment will be obtained to check these circuits.

9. Logging of the shale weighing system daily calibration has been initiated.

10. Extensive work has been performed on the liquid product system. Additional drains, heat tracing, insulation, and LACT pump actuating devices have

been added. Studies are being made to ascertain how to handle the sludge which accumulates with the water layer.

11. Designs are in progress to install valves under the 100-ton bin outlets. These will be used to stop shale and permit cleaning of the screening feeders under the bins.

    The potential of changing the screens in these feeders, from 1/4 inch bars to 1/2 or 3/4 inch, is being investigated. These would be used when testing 1 to 2 1/2 inch shale.

12. The Ty-lab building and their facilities have been completed and put into service.

13. The preventative maintenance program has been initiated in the crushing plant. This has proved effective.

14. Re-hardfacing of the primary jaw crusher and secondary roll crusher is in progress.

15. The ventilation system in the crusher plant is being completed. Problems have occurred with the water pump to the dust scrubber. The system, however, is very effective in operation.

16. The crusher plant apron feeder speed will be increased approximately 15% to put production rates at specifications.

17. Air manifolds were modified after the December 11-12 shutdown. The flanged joints in the 12 inch long spool pieces were removed and welded. Leaks had continued to occur.

18. Risers have bent during several runs. The most serious bending occurs when a hot clinker appears to wedge between the risers. Plans are to add additional gussets when changing shale sizes.

    During the last several shutdowns, no serious riser displacement has occurred even with clinkers. Any riser which has moved 3/8 of an inch is either reshaped or replaced on each shutdown.

C. Retort No. 3 Costs See Table 2.

1. Costs will approximate those shown in the December 15 Progress Memorandum. We are awaiting final

TABLE 2

RETORT NO. 3 - COST PROJECTION

Project Construction Budget - $725,000

		Stage II Scoping Study 2/28/66 Estimate	Torkelson Company May 1966 Estimate	Nov. 15 Estimated Final Cost Estimate	1/16/67 Estimated Costs
1.	Engineering and Construction Supervision	47,000	74,000	120,000	120,314.11*
2.	Materials - Equipment	373,000	346,000	405,000	405,000.00
3.	Construction and Construction Supervision	203,000	222,000	335,000	334,302.19*
4.	Contingency	51,000	38,000	--	--
5.	Fee	50,000	45,000	60,000	60,000.00
6.	Total	725,000	725,000	920,000	920,000.00

*Final Billing.

WSBergen
1/16/67

billing. Engineering and construction costs are final. Several invoices amounting to less than $5,000 total are expected soon.

2. Reviewing in more detail the construction overrun, $195,000, see Table 3. A report discussing both the engineering, construction, and costs will be completed by February 15, 1967.

D. Mine Grizzly

The mine grizzly was completed and shale processed for shipment and the Allis-Chalmers crushing test. Several revisions were required to bring the brizzly bars to specifications. Repairs and minor revisions were necessary as the screening progressed. This work is complete.

TABLE 3

RETORT NO. 3 - COST ESTIMATE

ANALYSIS OF BUDGET OVER-EXPENDITURE

Budget - $725,000
Estimated Final Cost - $920,000

1.	Field decisions necessitating additional engineering, construction, and equipment when detailed inspections showed existing equipment to be unsatisfactory for reliable and sustained operation. Estimated added cost	$ 67,000
2.	Process changes necessitating additional engineering, construction, and equipment. Estimated added cost	33,000
3.	Unusual delays in equipment delivery have resulted in extending the construction time one month and interrupting the continuity of the work effort. This condition resulted in additional overhead and construction costs. Estimated added cost	30,000
4.	Low cost estimate of (a) planned electrical work for $23,000 and (b) planned engineering cost for $27,000. Total added cost	$ 50,000
5.	Added fee due to higher job cost	15,000
	Total - Estimated added cost	$195,000

WSBergen
1/16/67

IV. RETORTING SECTION (J. F. Lawson)

A. Retorting Group (T. C. Lyons, W. M. Broman, R. L. Clampitt, P. H. Gifford, R. L. McGalliard, P. L. Reymond, E. F. Turner, J. W. Hasz)

1. Operation of Retort No. 3 - Run Series C-989 and C-990

Prior to Run Series C-989, the bolted connections on the air distributor assembly were welded to eliminate air leaks below the normal injection level (see Mechanical Engineering Section for details). The line burner was fired for Run C-989 on December 15, 1966; however, a failure of the recycle blower forced a shutdown before the unit was on run conditions.

Upon completion of the blower repairs, the unit was fired for Run Series C-990 at 0400 hours on December 24, 1966. The startup was smooth and the unit was on the target conditions about 27 hours later (0700, 12-25-66). The conditions were 400 lbs/(hr)(ft^2) mass rate, 4,500 SCF/T air, and 14,700 SCF/T recycle. The unit ran well at these conditions for about seven hours (until 1400 hours, 12-25-66). At this time, the bottom pressure began increasing rapidly and it rose from 7 inches H_2O to 10 inches H_2O in about 1 1/4 hours. Corrective action was taken when the pressure reached 10 inches H_2O. The recycle rate was reduced in 500 SCF/T increments. Five or six such cuts over a 30 minute period were made coupled with a 200 SCF/T decrease in air as the pressure continued to increase in a "sawtooth" fashion until it reached a level of 13 inches H_2O. At this point the unit "unloaded" as evidenced by a decrease in pressure.

The gas rates were brought back to conditions very slowly and the bottom pressure equilibrated at 6 to 6.5 inches H_2O. The unit ran smoothly for about nine hours after reaching conditions at which point the pressure began increasing in a similar fashion (6.5 to 9 inches H_2O in 1 1/4 hours). Recycle rates were cut and the pressure peaked below 10 inches H_2O. The recycle rate was held at 13,800 SCF/T for about eight hours (Run C-990-3) and then was increased to a new target of 14,300 SCF/T while holding the air at 4,500 SCF/T. The unit ran extremely smoothly at the new conditions for a 48 hour period following the pressure buildup (Runs C-990-3, 4, 5 and 6). The bottom pressure held just under 6 inches of H_2O with almost no fluctuation.

In light of this smooth operation, it is necessary to consider the possible reasons for the inability to operate for sustained periods at the higher gas rates (14,700 SCF/T recycle). The early operation was Christmas Day and the crushing plant was not operating. This necessitated drawing shale which was somewhat smaller in size than the desired Da of 1.55 inches and Dv of 1.75 inches from the 500 ton storage bin. Furthermore, this bin does not have facilities to make a final screening of 1/4 inch minus material prior to entering the retort. Therefore, it was believed that the smaller size and the greater fines concentration could have contributed to the gas rate limitation and a further test at the higher rates seemed advisable.

During the smooth operating period of December 26 and 27 the shale size returned to normal and at the end of the 48 hours the gas rates were increased once again to the previous level of 4,500 SCF/T air and 14,700 SCF/T recycle (0900, 12-28-66). The unit operation was very smooth for about 19 to 20 hours at which time the bottom pressure increased from 6 inches H_2O to 9.5 inches H_2O in 2 1/4 hours. As in the previous instances, the recycle rate was reduced to 13,000 SCF/T and this was effective in decreasing the unit pressures.

As a result of the third unsuccessful attempt to raise gas rates it was concluded that we had reached a true gas limitation and that our operation was close to the threshold. However, this conclusion is clouded by the fact that a storage bin charging the retort ran dry just prior to the upset and a "slug" of fines was undoubtedly charged to the retort. In any event, the observations of the control panel indicated that the unit recovered completely from the upset. (Runs C-990-7 and 8 were made during the high gas rate operation.)

Following the upset, the gas rates were returned to a lower level of 14,300 SCF/T recycle while continuing to hold the air at 4,500 SCF/T. These were the same rates used in runs C-990-3 through 6 which was a period of extremely smooth operation with high yields. The retort ran at these conditions for approximately six days (Runs C-990-9 through 19, December 29 through January 3) however, the overall operation was not as smooth as in the previous period in spite of the equivalent gas rates. The bottom pressure generally wandered over a range of 1 inch of H_2O (between 6 and 7 inches H_2O) and while this in itself is not bad, it did indicate a difference from earlier operation. More significant, however, was the fact that on three or

four occasions the bottom pressure increased to a level of 8 or 8.5 inches H_2O but the unit unloaded itself in each instance. It should be emphasized that no corrective action was necessary on the part of the operators to recover from these periodic pressure surges.

During this 6-day period at constant conditions, there were also several upsets of mechanical origin as opposed to those of a process nature. At 2020 hours on December 31 the top rotary feeder failed which shut down the raw shale feed to the unit. This necessitated a drastic reduction in recycle (to 5,000 SCF/T), air (to 900 SCF/T), and in the spent shale drawoff rate. The raw shale was off for about 1 1/2 hours. The following morning, the revolving distributor failed which stopped the raw shale feed to the unit for 3/4 hour. For another hour during repair, it was only possible to feed shale to one side of the unit. By all visual criteria, the unit recovered extremely well from both of these upsets.

During the last four balance periods of the 6-day, constant-condition operation (C-990-18 through 21), the spent shale drawoff system was operated at constant speed in an effort to smooth out the bottom pressure fluctuations. This was a rather minor change as the automatic mode of operation normally used holds the drawoff rate within narrow limits (± 5%). The manual or constant speed drawoff was abandoned at the conclusion of run C-990-21 since it did not appear to improve the operation.

In a continuing effort to investigate gas rates, the air was decreased from 4,500 SCF/T to 4,300 SCF/T while holding the recycle rate at 14,300 SCF/T (Runs C-990-20 and 21, January 4, 1967). This operation was carried out for a 24 hour period and there was no apparent improvement in operation as evidenced by the continuing mild fluctuation in bottom pressure. The gas rates were changed at 0800 on January 5 when a gradual pressure buildup to 10 inches H_2O required corrective action. The recycle rate was reduced to 10,000 SCF/T for about an hour and the pressure returned to the normal level. The observations made during this upset indicated that it was somewhat different from the pressure buildups noted previously. The location of the maximum pressure gradient was observed higher in the bed indicating a "liquid rich" condition as compared to a "dust rich" condition which generally occurs just above the air distributors. As

in the previous upsets, the unit appeared to recover completely.

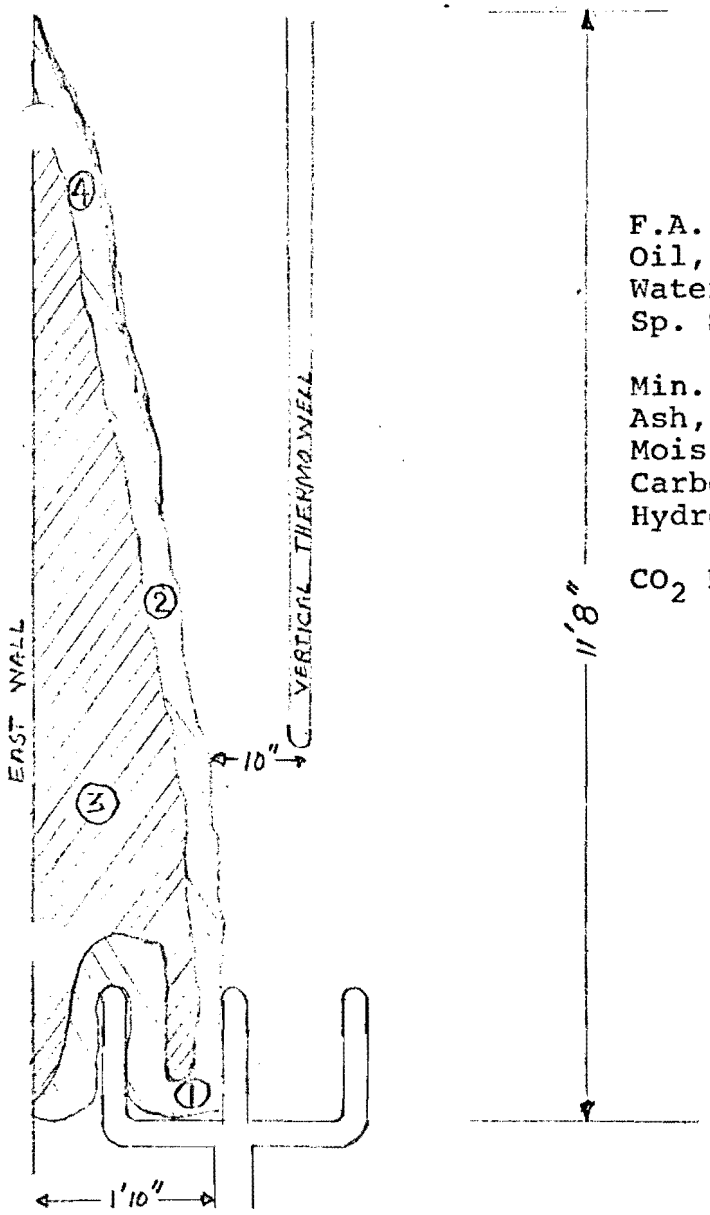
After the recovery, the unit was lined out at a new set of gas rates: 4,500 SCF/T air and 14,000 SCF/T recycle. Although this change was in the direction of lower yields, it was made in an effort to determine the price of obtaining a smooth surge-free operation. The operation was smooth for about 36 hours (until 1700 hours, 1-6-67), at which time a storage bin was emptied inadvertently and this was followed by a characteristic buildup in bottom pressure. Following the corrective action the unit was lined out on conditions and ran smoothly for an additional 17 hours with the exception of one instantaneous pressure surge. Shortly after noon on January 7, one of the four offgas temperatures increased about 50 F. This was the first clear-cut evidence that the unit was in serious difficulty. The split in offgas temperatures was followed by a general cooling-off of the west half of the retort. The usual corrective action - cutting gas rates for several hours - did not improve the operation. The unit temperatures and a shale flow check indicated a clinker in the eastern section of the retort; consequently, the unit was shutdown at 2100 hours on January 7. The subsequent inspection revealed a clinker positioned as shown in Figure 1. This final series at the lower gas rates included Runs C-990-22 through 26.

A detailed analysis of the unit data was made in a joint effort with the Engineering Analysis Group. Although the most probable cause of the shutdown was the "slug" of fines introduced on the evening of January 6, there were indications that the unit was in trouble prior to this event. Some suspicious shifts in unit temperatures indicated that the clinker may have formed during the night of January 5 or the day of January 6 which is prior to the upset associated with the empty bin on the evening of the 6th. In any event, positive steps are being taken to minimize the charging of large "slugs" of fines during future operation.

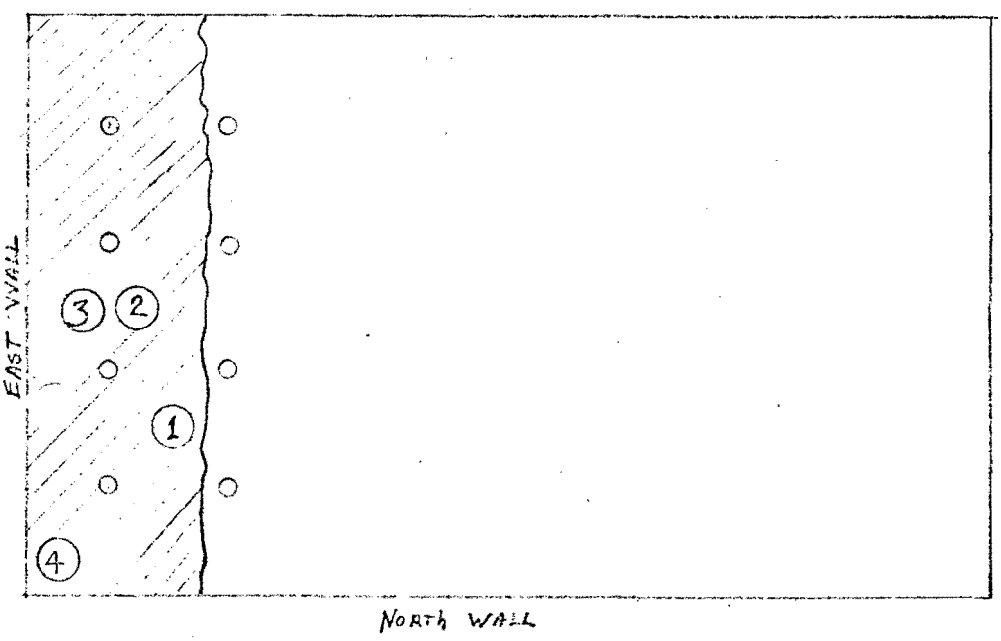
It should be emphasized that the gas rates discussed up to this point were targets which were used to adjust the operation of the unit from day to day. The rates in SCF/T assume that the shale mass rate will be as specified and any deviation in the mass rate or molecular weight of the gas streams will change the values in the final computer calculations. Thus, the gas rates discussed in the following section will vary from the target values presented up to this time.

FIGURE 1

CLINKER FOUND ON SHUTDOWN - RUN C-990



	①	②	③	④
F.A., GAL/T	0.3	0.1	0.3	10.2
Oil, Wt%	0.1	0.1	0.1	3.9
Water, Wt%	0.2	0.3	0.2	0.7
Sp. Shale, Wt%	99.7	99.4	99.3	94.4
Min. CO_2, Wt%	1.26	17.3	12.7	19.0
Ash, Wt%	99.2	79.6	85.0	72.0
Moisture, Wt%	0.02	0.02	0.02	0.01
Carbon, Wt%	0.54	6.46	2.56	12.5
Hydrogen Wt%	0.54	0.20	0.07	0.85
CO_2 Decomp.	95	13	40	-5



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2. Preliminary Data Analysis - Retort No. 3

The yield data from the shakedown operations on Retort No. 3 should still be considered preliminary. As discussed in the operations section of this report, several adjustments were made in gas rates to determine their effect on retort operability. The results from these test runs are given in Table 4. Pertinent yield data are given below:

Actual Gas Rates

Run Nos.	Shale Rate, lbs/(hr)(ft^2)	Air Rate, SCF/T	Recycle Rate, SCF/T	Yield, Vol % FA
C-990-6 thru 9	393	4,540	14,540	87.3
C-990-7 and 8	389	4,640	15,340	90.5
C-990-9 thru 19	387	4,650	14,790	87.2
C-990-20 and 21	399	4,350	14,360	85.0
C-990-22 thru 25	400	4,500	13,700	81.8

The target conditions for runs C-990-3 through C-990-6 and runs C-990-9 through C-990-19 were the same. The average yields for both sets of runs were essentially the same (87.3% and 87.2%). The average yield for C-990-7 and C-990-8 was 90.5%. These runs were attempts to duplicate the original conditions used in the first tests (PTC-990 and C-990-2) of this run series. Pressure buildups in the retort required a decrease in recycle rate.

Runs C-990-20 and C-990-21 were made at actual conditions of 4,350 SCF/T air and 14,360 SCF/T recycle gas and the average yield was 85.0%.

Runs C-990-22 through C-990-25 were made at actual gas rates of 4,350 SCF/T air and 13,700 SCF/T recycle and the average yield was 81.8% of Fischer Assay. Run C-990-26 was not included in this average because of poor operability.

A cursory examination of the data from Run Series C-990 indicates a large effect of recycle gas rate on yield. The yield was 81.8% with 13,700 SCF/T recycle gas rate and a yield of 87.2% with a recycle gas rate of 14,650 SCF/T.

As mentioned previously the yields from the shakedown runs (C-988 and C-990 series) should be considered preliminary. Improvements were made in the various accounting and sampling systems before and during Run C-990.

TABLE 4

RESULTS FROM SHAKEDOWN OPERATIONS ON RETORT NO. 3 RUN SERIES C-990

Run No.[1]	PTC-990 and C-990-2	C-990-3 through C-990-6	C-990-7 and C-990-8	C-990-9 through C-990-19	C-990-20 and C-990-21	C-990-22 through C-990-25	C-990-22 through C-990-26
Shale Size, inches	1 - 2 1/2	1 - 2 1/2	1 - 2 1/2	1 - 2 1/2	1 - 2 1/2	1 - 2 1/2	1 - 2 1/2
Da	1.311	1.427	1.538	1.465	1.590	1.525	1.514
Dv	1.593	1.659	1.774	1.719	1.793	1.716	1.706
Shale rate, lbs/(Hr)(Ft^2)	405	393	389	387	399	400	399
Actual Air Rate, SCF/T	4,430	4,540	4,640	4,650	4,350	4,500	4,480
Actual Recycle Rate, SCF/T	14,000	14,540	15,340	14,790	14,360	13,700	13,700
FA, GAL/T	25.4	24.6	25.0	25.9	26.9	26.2	26.1
Offgas Temperature F[2]	125	125	125	126	127	125	125
Spent Shale Temp. F[2]	365	362	363	376	379	371	372
Retort Pressures, in/ft							
Overall	0.38	0.30	0.32	0.32	0.33	0.33	0.32
Above A/D	0.48	0.35	0.34	0.36	0.36	0.32	0.32
Maximum	2.0	1.2	1.2	1.3	1.3	1.2	1.2
Yield, Vol,%	83.1	87.3	90.5	87.2	85.0	81.9	80.8
Spent Shale, GAL/T	3.1[3]	0.5	0.5	0.4	0.3	0.6	0.9
CO_2 Decomposition, Wt%	28.9	27.5	30.5	26.8	22.0	24.7	23.3
Material Balances, Wt%							
Overall	100.0	100.0	101.4	102.0	100.7	101.2	101.2
Organic Carbon	103.8	105.6	105.4	105.9	102.1	102.0	102.4
Organic Hydrogen	101.8	102.9	106.6	94.5	91.7	90.0	90.1
Water	109.0	97.9	121.8	158.8	156.9	151.7	149.1
Spent Shale Control	Mode 2	Mode 2	Mode 2	Mode 2	Manual	Mode 2	Mode 2
Operability	Recycle gas rate had to be reduced. Operations rugged until gas reduced.	Recycle rate had to be reduced again. Operations good.	Fair. Recycle gas rate had to be reduced from 14.700 at end of C-990-8.	& Manual Fair to good.	Fair to good.	Fair.	Fair except for C-990-26, which was poor.

(1) Averaged data.

(2) Temperatures probably 10 F low - TR5 recorder found to be reading low - Low also for C-988.

(3) PTC-990 only.

Note:

Target Conditions	Shale Rate lbs/(Hr)(Ft^2)	Air Rate SCF/T	Recycle Rate SCF/T
PTC-990, C-990-2, C-990-21, C-990-8	400	4,500	14,700
C-990-3 through C-990-6, C-990-9 through C-990-19	400	4,500	14,300
C-990-20, C-990-21	400	4,300	14,300
C-990-22 through C-990-26	400	4,500	14,000

EETurner
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Presented on Figure 2 is the material balance history for Run C-990-1 through 26. This figure shows the type of fluctuations in the results. In reviewing Figure 2, it should be remembered that various accouting systems are in the process of being revised in order to improve the quality and consistency of all future raw data. Raw shale, spent shale, and liquid sampling problems along with other accouting problems are responsible for the very high organic carbon, overall and water balances on Figure 2. The following program is in progress to correct these problems.

(1) Improve the raw shale accounting system by eliminating any inconsistency in measurements between the Merrick scale and the Hardy scale on the weigh bin.

(2) Insure good raw shale and spent shale sampling.

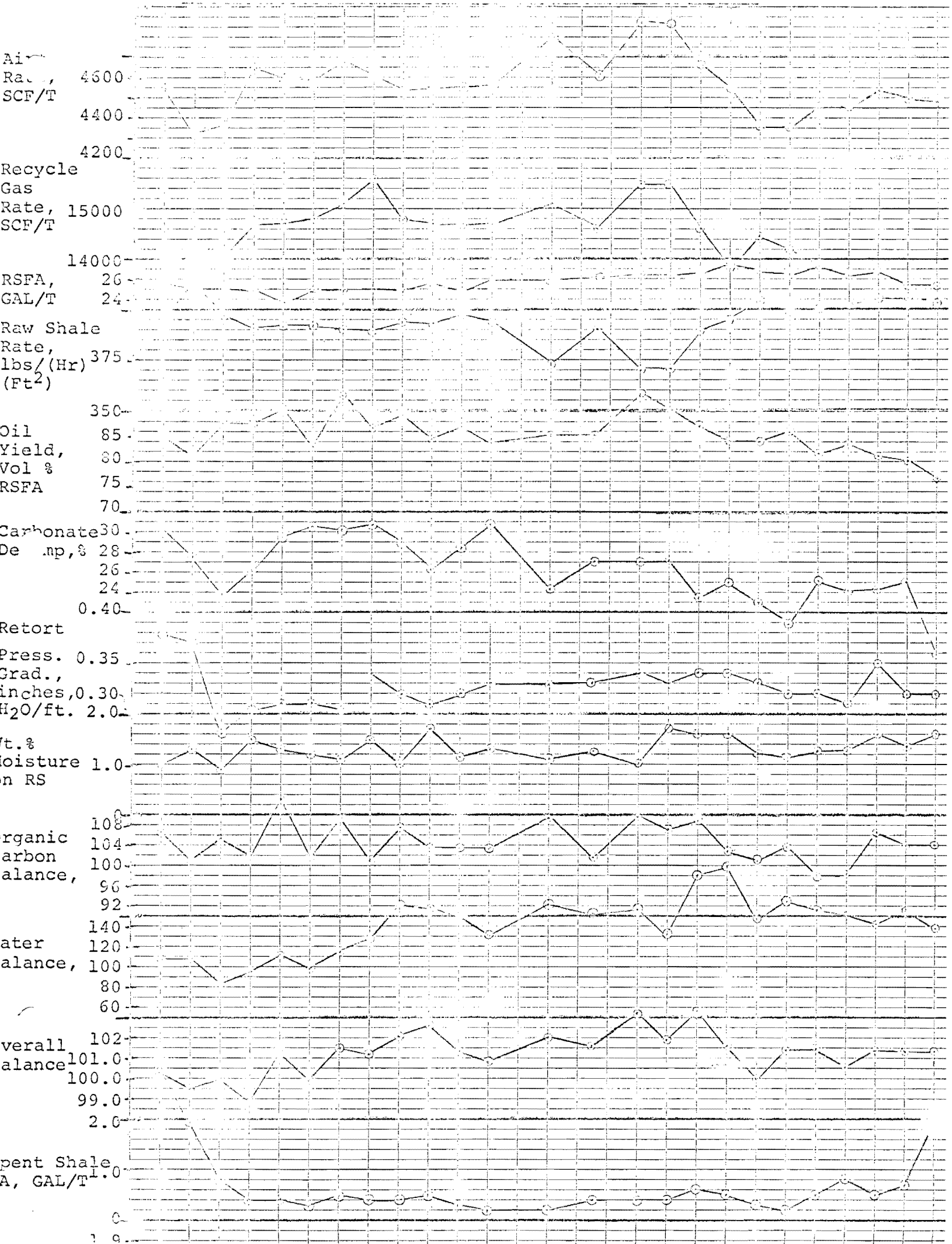
(3) Improve the oil-water separation in the liquid recovery system.

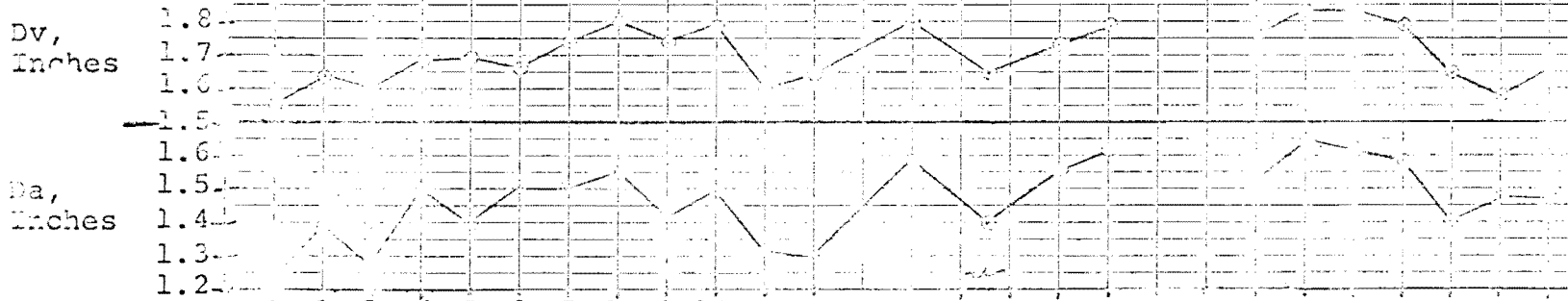
In general, the process operability of Run C-990 was fair to good. The judgment on operability during this shakedown run was aggravated by happenings not necessarily germane to process operability. During the run, gas rates were changed several times to determine the upper limits of gas rates on operability. These studies plus inadvertent addition of fines, freezing of instrument lines, and "others" tend to make operabilit determination more a matter of judgment. There can always be a question about the condition inside the retort after a severe upset due to excess gas rates or fines being inadvertently added to the unit, or some mechanical failure that stopped shale flow in the retort. Because of the foregoing factors, judgments with regard to the quality of operation during short periods are difficult and subject to change, and, therefore, should be interpreted with caution.

The average shale size distribution for Run C-990 is given in Figure 3. The average Da and Dv of the shale during the run were very close to the estimated commercial fraction. The Da was 1.46 inches as compared to the estimated commercial Da of 1.55 inches. The Dv was 1.71 inches as compared to 1.75 inches. The 98% points were essentially the same and the 5% point was 0.74 inches for the run as compared to 0.86 inches for the estimated commercial shale. The shale used during the run has approximately 1 1/2% material less than 1/4 inch as compared to none for the estimated commercial type shale.

FIGURE 2

MATERIAL BALANCE HISTORY FOR RUN C-990 1 THROUGH 26





Material Balance Number

Run C-990-1 through 26 Retort No. 3

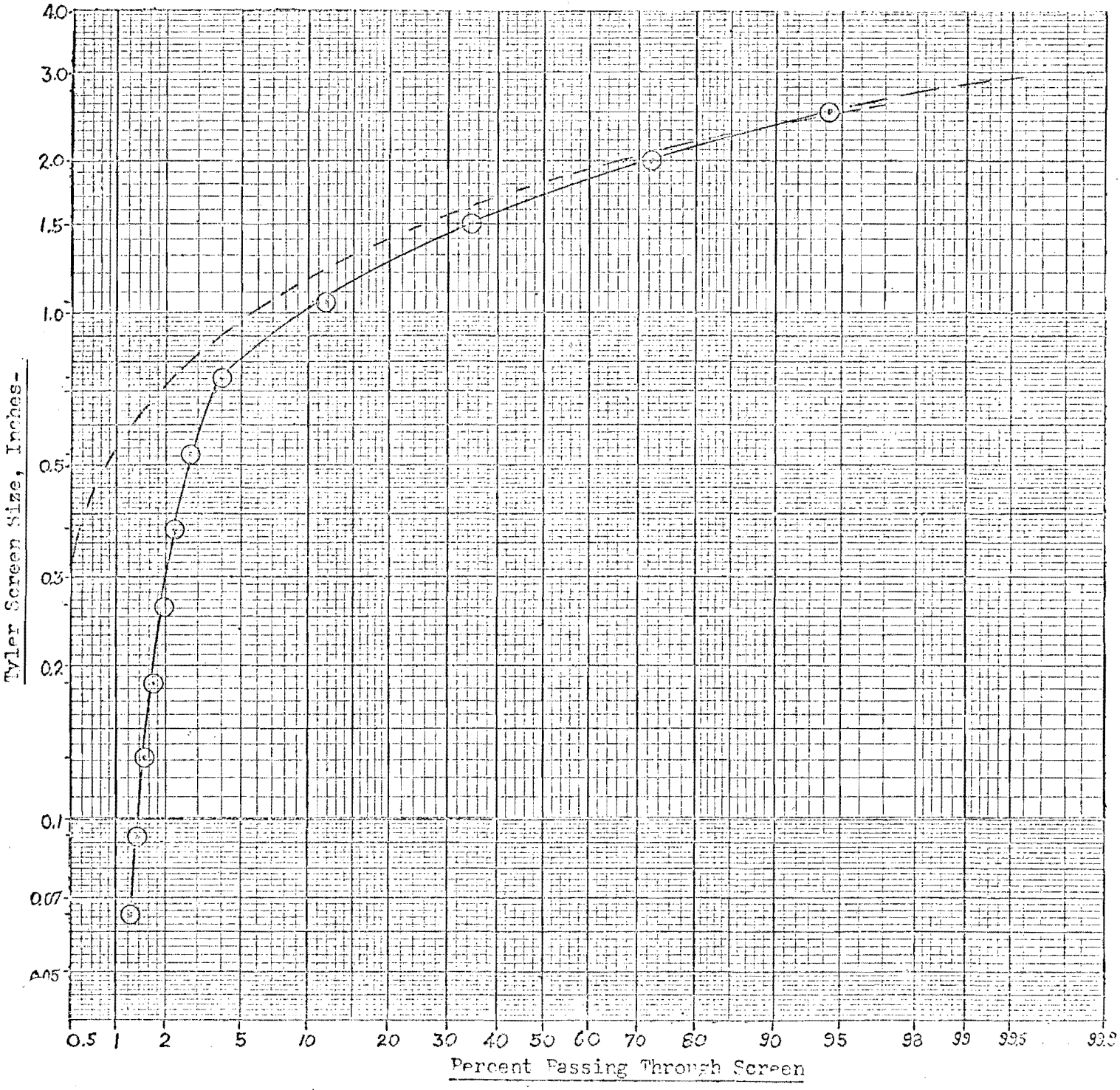
RLClampitt

FIGURE 3

SHALE SIZE DISTRIBUTION

	Da	Dv
⊙——⊙ Run C-990-1 Through 26	1.46	1.71
– – – – Estimated Commercial Fraction	1.55	1.75

·1 to 2 1/2 inch Shale
Retort No. 3
Time Interval - 12/25/66 to 1/7/67



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3. Temperature and Pressure Profile Comparisons For Retorts No. 2 and No. 3

Figure 4 shows a comparison between the average temperature profiles for B-952 tests E through L on Retort No. 3 and runs C-988 and C-990 on Retort No. 3. The C-988 curve is made up of test periods 3 through 6 which seem to be typical of this run series. C-990 has been separated into two groups. One group is made up of test periods 1, 2, 7 and 8 in which the target recycle rate was 14,700 SCF/T. The other group is made up of test periods 4, 5 and 6 in which the target recycle was 14,300 SCF/T.

There is not much difference in the shapes of the temperature profiles above the air distributors. The difference in heights of runs C-988 and C-990 is explained by the higher recycle rate in run C-988. Comparing run C-988 with B-952, C-988 is somewhat higher even though the recycle rates were approximately the same. This shows that Retort No. 3 is somewhat more sensitive to gas rates than Retort No. 2. This relates to the difference in the area configuration of the two retorts due to the presence of the liner in Retort No. 2 which ended 8 inches above the air inlets. The net result is somewhat higher gas velocities in the combustion zone in Retort No. 3 at equivalent conditions.

The temperature profiles below the air distributors for C-988 and C-990 are identical in shape and again the difference in heights can be explained by the difference in recycle. When comparing either of these with B-952 some caution should be observed. The temperature points used for B-952 were from skin thermocouples while those in C-988 and C-990 were from vertical thermowells. The reason for the odd shape of curve B-952 is probably due to wall effects. Curve B-952 does seem to be somewhat hotter than C-988 or C-990.

In both C-988 and C-990 after 7 feet below the air distributors the shale begins to heat up again. This is probably the shale coming to an equilibrium state. The surface of the shale cools when it passes through the recycle gas stream then the center of the shale heats it back up again when it leaves this stream. The equilibrium temperature would then be about the temperature of the spent shale.

In Figure 5, there is a comparison between the average pressure profiles for B-952, C-988 and C-990. The test

FIGURE 4

VERTICAL TEMPERATURE PROFILE

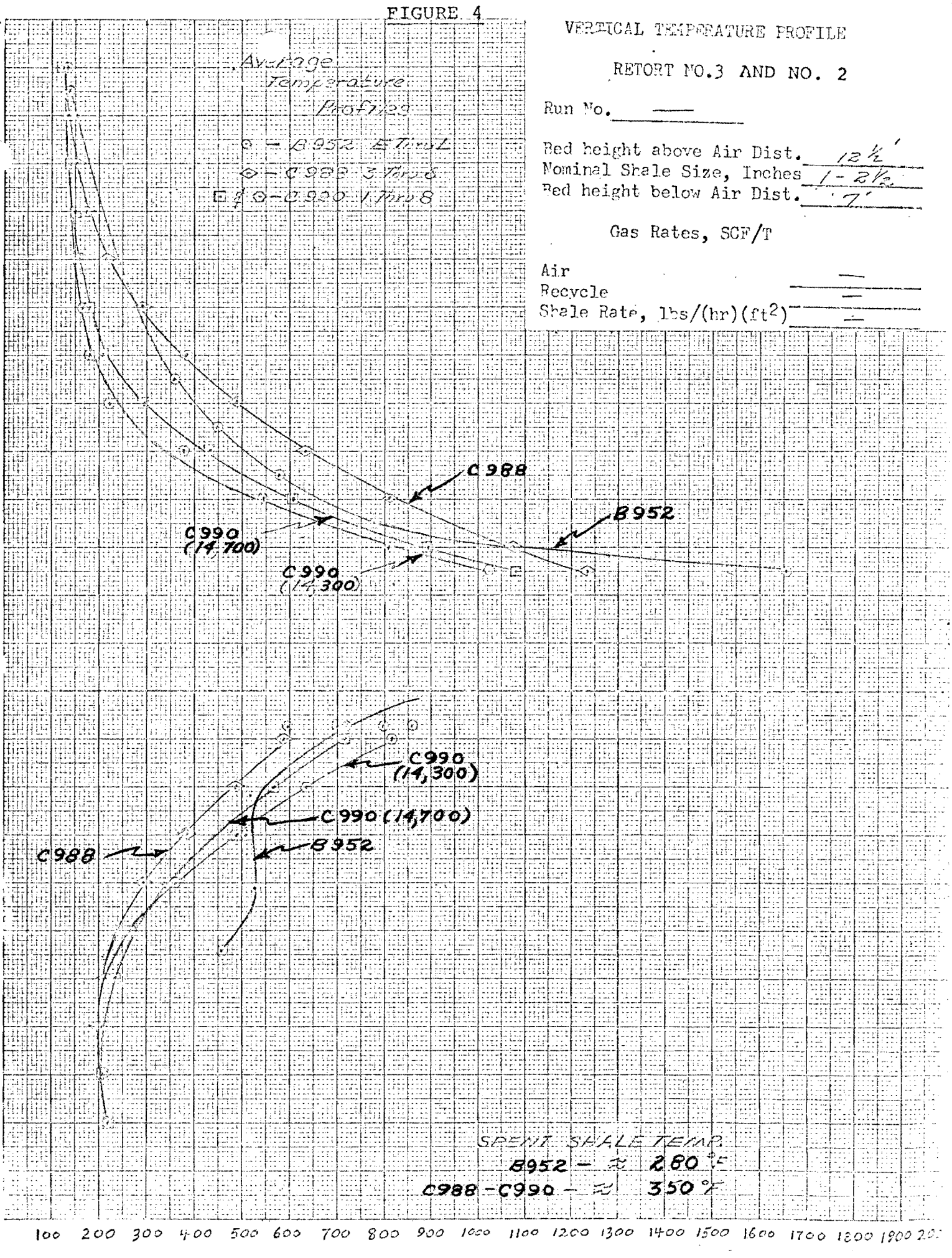
RETORT NO.3 AND NO. 2

Run No. ——

Bed height above Air Dist. 12½'
Nominal Shale Size, Inches 1 - 2½
Bed height below Air Dist. 7'

Gas Rates, SCF/T

Air —
Recycle —
Shale Rate, lbs/(hr)(ft²) —

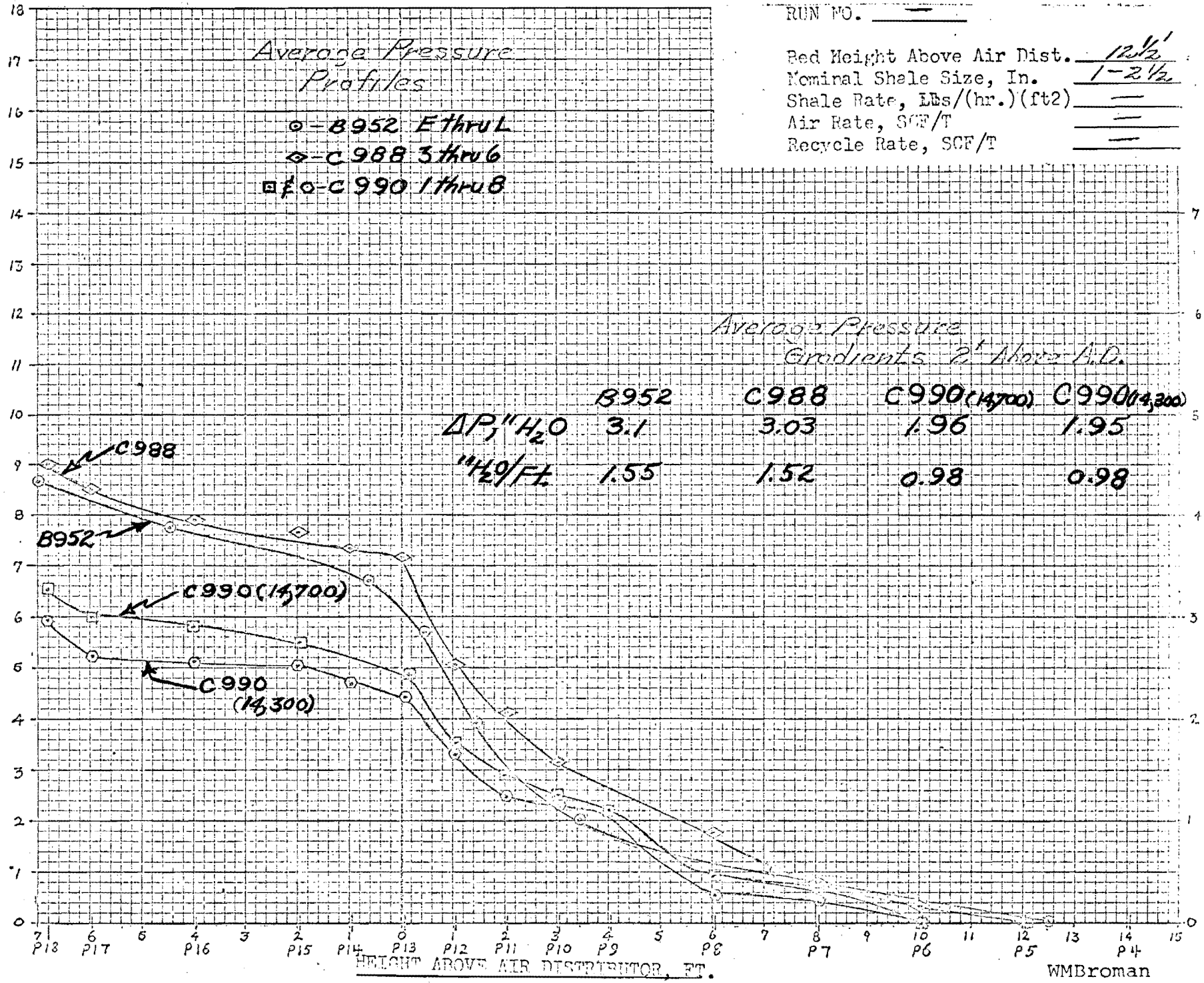


TC-1= TC-3=

WMBroman
1/3/67

FIGURE 5

VERTICAL PRESSURE PROFILE - RETORT NO. 3 AND NO. 2



WMBroman
1/3/67

periods used in the averages were the same as those used in the temperature profiles. The reduction in reducle rate from 15,000 SCF/T in C-988 to 14,300 - 14,700 SCF/T in C-990 resulted in a considerably lower bottom pressure (9 inches H_2O in C-988 to 6 - 6.5 inches H_2O in C-990) and pressure gradient (1.52 inches H_2O in C-988 to 0.98 inch in C-990). Comparing C-988 with B-952, the pressure gradient two feet above the air distributors is about the same, but for the same recycle rate the profile for C-988 is higher than B-952. This also shows that Retort No. 3 is more sensitive to gas rates than Retort No. 2.

B. Mechanical Model Studies (L. J. Skowronek)

1. Retort No. 3 (1/6 Scale Model) Studies

The model is being used to screen the air distributor configurations planned for use in retorting 1/4 to 1 inch and 1/4 to 2 1/2 inch range shales. The present contemplated arrangement calls for 54 and/or 45 riser assemblies.

The studies are now in progress and will be reported on in the next Monthly Progress Memorandum.

2. Retort No. 3 (Half Section Model) Studies

The model has been reconditioned and modified to reflect the proposed air distributor assembly for retorting 1/4 to 1 inch shale (54 riser configuration). Work in developing flow patterns will be reported in the next Monthly Progress Memorandum.

3. Corrosion-Erosion Program

The monthly corrosion-erosion report was completed with transmittal to the participants on December 29, 1966.

During the unit shutdown of January 7, 1967 the unit was inspected in detail and corrosion-erosion measurements were made on all the unit assemblies. These along with data taken on mine handling equipment will be reported in the monthly corrosion-erosion memorandum.

4. Recycle Gas Distributor Study

The model to study recycle gas distribution has been reconditioned and the internals have been installed. As soon as the press of Retort No. 3 work eases, the tests will be performed.

C. Bench Scale Studies (D. P. Cotrupe)

1. Gas Flow Studies

As reported in the December Monthly Progress Memorandum, preliminary results indicated that the problem of recovering oil mist from the offgas stream had been solved by installation of a glass wool-packed column in the recovery train. Further work has shown that this was not the case. High pressure drops (6 to 7 inches Hg) were obtained across the glass wool column and in order to maintain near-atmospheric pressure at the top of the retort, it was necessary to operate the low temperature condensers downstream of the packed column under a vacuum of 6 to 7 inches Hg. The efficiency of the low temperature condensers was significantly reduced under these conditions, resulting in losses of water and lighter oil products and in serious plugging of the lines in the -110 F condensing system.

Several modifications were made on the packed column and alternate operating procedures were tested in an effort to reduce the pressure drop while maintaining recovery efficiency. None of these actions was completely successful for in each case, although the pressure drop was reduced to an acceptable operating level, the efficiency of the unit was also reduced, resulting in mist carryover through the recovery train. It is apparent from this work that the present recovery system is inadequate for the proposed gas flow studies and would be so for any future bench scale retorting studies which may be contemplated. Therefore it was decided that work on gas flow studies be temporarily discontinued until an adequate mist recovery unit (e.g. a small electrostatic precipitator) is purchased or constructed.

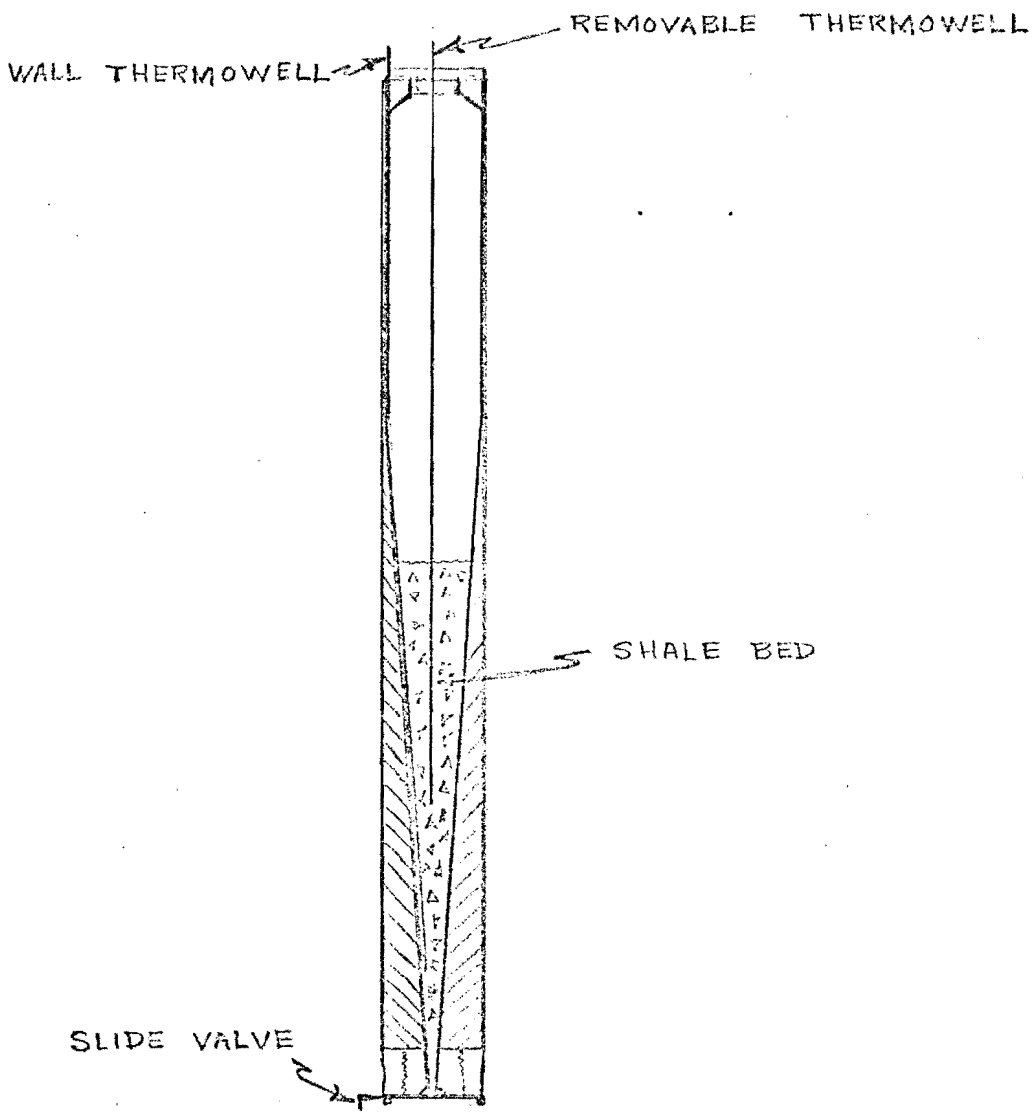
2. Heated Shale Flow Studies

An item of considerable interest, particularly with respect to retort operability, is the effect of temperature on the "flowability" of shale. Therefore, a short program has been designed and is in progress to study the flow characteristics of shale at elevated temperatures. The studies are being carried out in the Mini-Retort which has been equipped with a slide valve as shown in Figure 6. Briefly the operating procedure involves charging shale to the preheated unit, heating the shale to a specified temperature, discharging the shale from the bottom of the unit by means of a slide valve, and measuring the time required to discharge the shale. In practice, the retort is preheated to the

FIGURE 6

SKETCH OF MINI-RETORT AS USED IN

HEATED SHALE FLOW STUDIES



WEIGHT OF SHALE CHARGE : 2.2 lb

VOLUME " " " : 0.03 FT^3

HEIGHT " " BED : 1.6 FT

DIAMETER OF OUTLET : 0.5 IN.

P.P. COTRUPS

1-13-67

desired temperature level, the shale is charged and heated as rapidly as possible to this level. Extreme care is taken to insure that no measured temperatures exceed the specified level. When the entire bed has reached the operating temperature level, the center thermowell is removed, the slide valve opened and the discharge time measured.

To date, shale flow has been investigated at temperatures ranging from 80 to 1000 F using 8 to 14 mesh, 28 gallon per ton raw shale. The experimental program was designed to investigate temperature increments of 200 F beginning at ambient temperature and continuing upward until a discontinuity in flow was observed. Temperatures above this level would also be examined to determine if and at what temperature continuous flow is reestablished. The experimental results obtained thus far appear in Table 5 and are plotted in Figure 7. The discharge times reported in the table are averages of at least three runs made at the same conditions.

As indicated in Table 5, the flow at 200 and 400 F was extremely smooth. In both cases the discharged shale was not visibly different than that charged, although a weight loss (presumably water) of $\sim$ 0.5% was obtained in the 400 F runs. At the 600 F level, several differences were observed in addition to the significant loss in flow efficiency. Shale flow did not commence immediately upon opening of the slide valve; however continuous flow was readily established by tapping the bottom of the retort. Once started, the flow was very smooth and the retort walls were clean after each test. A small amount of water and oil, equivalent to $\sim$ 1 Wt % of the charge, were distilled off during the heating process and as expected, a large number of the discharged shale particles were blackened.

At 800 F, continuous flow could not be established. The bed was apparently completely bridged since it was necessary to continuously tap the retort to discharge the shale. Slugs of shale were removed with each tap but at no time did the shale flow without tapping. A large fraction of the discharged shale was in the form of shale agglomerates rather than discrete particles, suggesting the existence of the "cohesive mass" to which reference has often been made in analyses of retort performance. The observed flow characteristics at these conditions would also support this theory. It should be noted that a considerable amount of rerorting had taken place during the heating process. Surprisingly, the retort walls were clean and completely free of deposits of any kind after these runs.

TABLE 5

EFFECT OF TEMPERATURE ON SHALE FLOW

Raw Shale Assay : 28 GAL/T
Volume Shale Charge: 0.03 Ft^3
Diameter of Outlet : 0.5 Inches

Temperature, F	Flow Time, Sec. Range	Flow Time, Sec. Average	Flow Rate, Ft^3/min.	Flow Factor, t_{800F}/t_{T^oF}	Remarks
80	16.2-16.4	16.3	0.110	1.00	Smooth flow, clean retort walls.
200	16.6-17.2	16.9	0.106	0.964	Smooth flow, clean retort walls.
400	16.8-17.0	16.9	0.106	0.964	Smooth flow, clean retort walls.
600	18-20	19	0.095	0.857	Tap start, smooth flow, clean retort walls.
700	22-24	23	0.078	0.709	Tap start, smooth flow, clean retort walls.
800	--	--	0	0	No flow, required continuous tapping to discharge shale, clean walls.
1000	--	--	0	0	No flow, required rodding to discharge shale, heavy shale deposits on retort walls.

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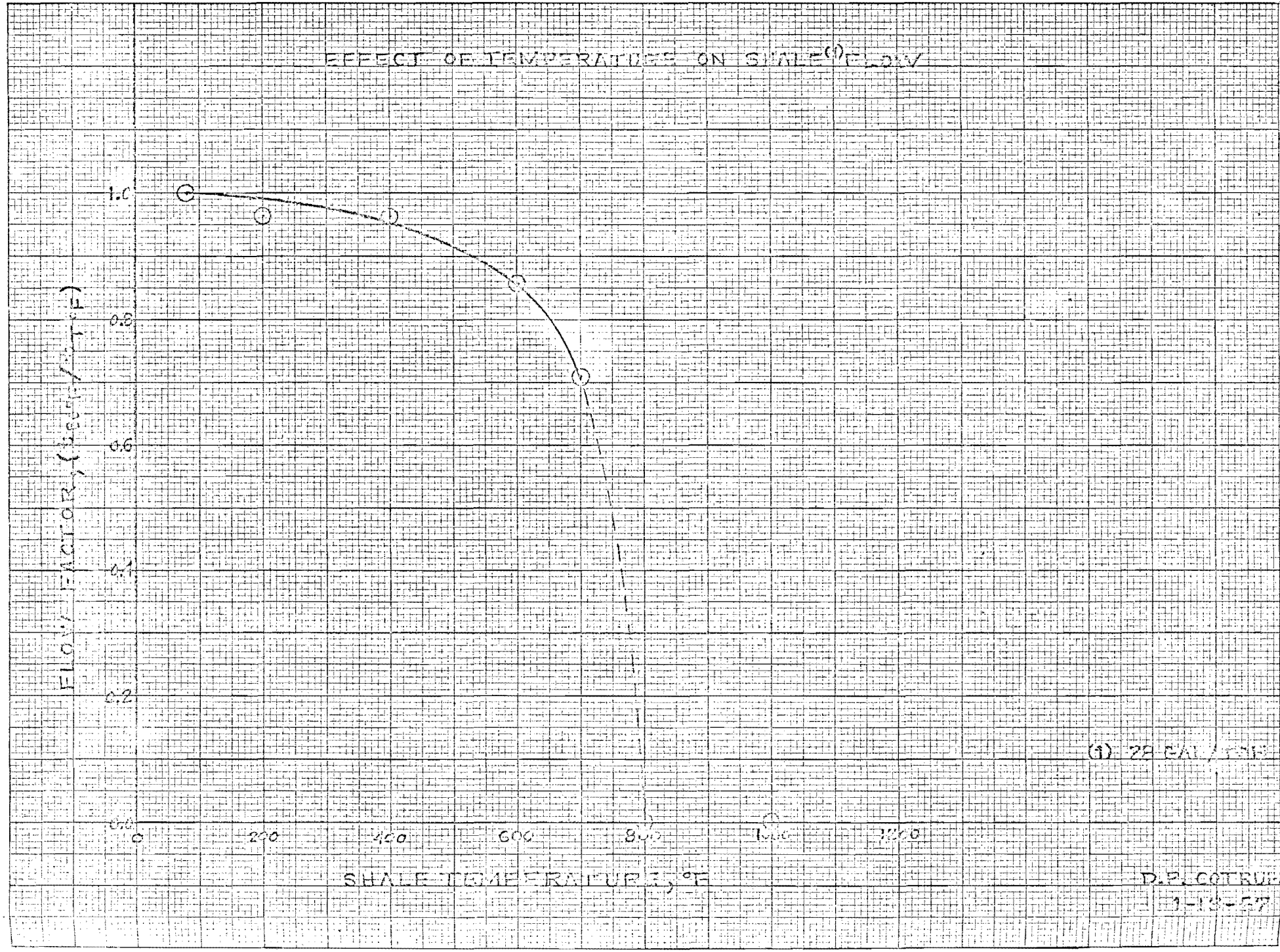
FIGURE 7


EFFECT OF TEMPERATURE ON SHALE(1) FLOW
FLOW FACTOR, (lbs/hr-ft²-°F)
1.0
0.8
0.6
0.4
0.2
0.0
0
200
400
600
800
1000
1200
SHALE TEMPERATURE, °F
(1) 22 GAL/TON
D.P. COTRUVE
1-12-57

Following the 800 F runs, tests were made at 1000 F to determine whether flow could be reestablished by drying out the shale after retorting is complete. The shale did not flow. In fact, the retort could not be emptied by tapping as in the 800 F runs, rather it was necessary to "rod" the retort to discharge the shale. Again the discharged material contained a large fraction of shale agglomerates. Further, the retort walls were heavily deposited with shale. Apparently the agglomerates formed during retorting became coked and hardened at the higher temperature.

The tests at 700 F were made to better define the point of discontinuity in the shale flow curve. As shown in Figure 7, the flow was $\sim$70% as fast as that at 80 F. Water and oil equivalent to $\sim$ 2 Wt % of the original charge was distilled off during the heating cycle. In other respects, these runs were similar to those made at 600 F in that tap starts were required, but once started, the flow was very smooth. The retort walls were clean after these runs also.

V. ENGINEERING AND ECONOMIC ANALYSES (K. I. Jagel, Jr. and R. A. Reitz)

A. Allis-Chalmers Primary Crushing Test

Approximately 200 tons of oil shale were shipped on schedule for the Allis-Chalmers primary crushing test in Florida. However, the crushing test has been postponed to January 21 because of problems with the rail cars. An expected late delivery of the rail cars plus an on-route car repair forced the postponment from January 14.

A 6"$^+$ mine run feed was prepared for the test with the new mine run grizzly. As is to be expected, degradation was obtained from the grizzly and from handling, but the material shipped is far superior for testing compared to 200 tons of mine run. The 6"$^+$ shale will be crushed in a 36"ø X 60" Fairmount single roll tooth type crusher to obtain crusher capacity, product size distribution, and energy required for crushing.

B. Comparison of Material Balance Results for Retort No. 3 With Retort No. 2 Demonstration Run Results

The following table is a comparison of the material balance results obtained in Run B-817C-U in Retort No. 2 with those obtained in Run C-990 which was recently completed in Retort No. 3.

	Retort No. 2 Run B-817C-U		Retort No. 3 Run C-990-1 to 26	
	Mean	95% C.L.	Mean	95% C.L.
Overall	98.7	0.8	101.2	2.2
Organic Carbon	96.0	5.0	104.6	7.8
Organic Hydrogen	99.2	7.0	96.3	14.9
Water	80.3	10.0	140.0	58.9
Oxygen	96.3	2.5	106.9	9.4

Apparently, the precision of the Retort No. 3 material balances is significantly poorer than that obtained in Retort No. 2 during excellent operation. W. M. Broman of the Retort Group has uncovered one source of error that seems important. Spent shale dust rises by natural conviction up the raw shale splitter waste duct and appears to contaminate the raw shale sample. This type of contamination would cause a high estimate of the spent shale rate and would adversely affect at least the overall and organic carbon balances. This problem will be corrected in the near future. A second area of concern is the flow rate through the gas sampling system. The flow meter in this system has not yet been calibrated. Another source of

possible error that should be checked is the water rate from the decanter. In the original design, a water meter was provided. It has been found impractical to operate this meter because of the solids in the water. Water rate is now determined by tank gauging. This tank has not been calibrated.

C. Evaluation of Raw Shale Rate Control Instrumentation On Retort No. 3

The raw shale rate to Retort No. 3 is controlled by the Merrick Weight-O-Meter. In order that the set point for this rate controller can be precisely read, a digital voltmeter has been provided.

In this study, the hourly shale rate was noted for a number of hours at various set point locations near 12 tons per hour. This study has shown that the relationship between set point voltage and rate is linear, as expected, and that the standard deviation of the output at a fixed set point is ±0.16 tons per hour at the 12 tons per hour rate. The 95% confidence interval of the output at this same rate is ±0.32 tons per hour or about ±2.7%.

The tabulated data for this study is presented in Table 6.

D. Material and Heat Balance Program for Retort No. 3

The computer program for calculating material and heat balances on Retort No. 3 has been adapted from that previously used for Retort No. 2. This adaptation of the program has been in use since November 22, 1966 and all runs have been calculated using it. A memorandum will be written which will describe the source of each of the input items on the input sheet for this calculation.

E. Carbonate Decomposition Time at Temperatures in the Air Distributor Region

After the shutdown of Run C-990, a portion of the clinker observed on the east side had a 95% carbonate decomposition. This sample was taken above the air distributor manifold near the bottom of the clinker. The question has been asked whether this could happen at the 1100 to 1200 F temperatures which may have existed in this region. The relationship between carbonate decomposition and time at temperatures of 1100 and 1200 F is shown in Figure 8. At 1100 F, 50% carbonate decomposition occurs in about 1.4 days and 95% in 5.5 days while at 1200 F, 50% carbonate decomposition occurs in 0.15 days and 95% in 0.65 days. The temperature profile anomalies that have been observed preceding this shutdown started to occur about two days before the shutdown. It

TABLE 6

EVALUATION OF RAW SHALE RATE CONTROL INSTRUMENTATION

Set Point DVM Reading, Mv.	Raw Shale Rate, TON/HR	Set Point DVM Reading, Mv.	Raw Shale Rate, TON/HR
380.0	11.41	407.5	12.06
	11.17		11.97
	11.22		12.06
	11.30	407.7	12.03
	11.34	407.8	12.05
402.0	12.09		12.04
	11.85		12.06
	11.87		12.04
	11.84		12.11
403.2	11.90		12.01
	11.82		12.06
	11.90		11.94
	11.92		12.12
	11.85		11.96
	11.91	410.0	12.17
	11.90		11.93
403.4	11.85	410.2	12.18
	11.91		11.16
403.6	11.95		12.08
	11.80		12.24
	11.84	411.5	12.40
	12.00		12.14
403.8	11.85		12.05
	11.91		12.18
404.0	11.74	412.2	12.00
	11.77		12.11
	11.81		12.03
	11.94		12.08
404.2	11.86	412.3	12.26
	11.89		12.20
	11.92		12.16
	11.88	412.4	12.10
	11.83		12.18
405.6	11.67		12.15
405.8	12.05		12.16
	12.40		12.18
	12.36		11.90
406.0	12.27		12.20
407.3	12.00		12.21
	11.95	413.0	12.30
	11.98		12.18
	11.98		12.18
	11.98		11.96
	11.98		12.17
407.5	12.07		12.15

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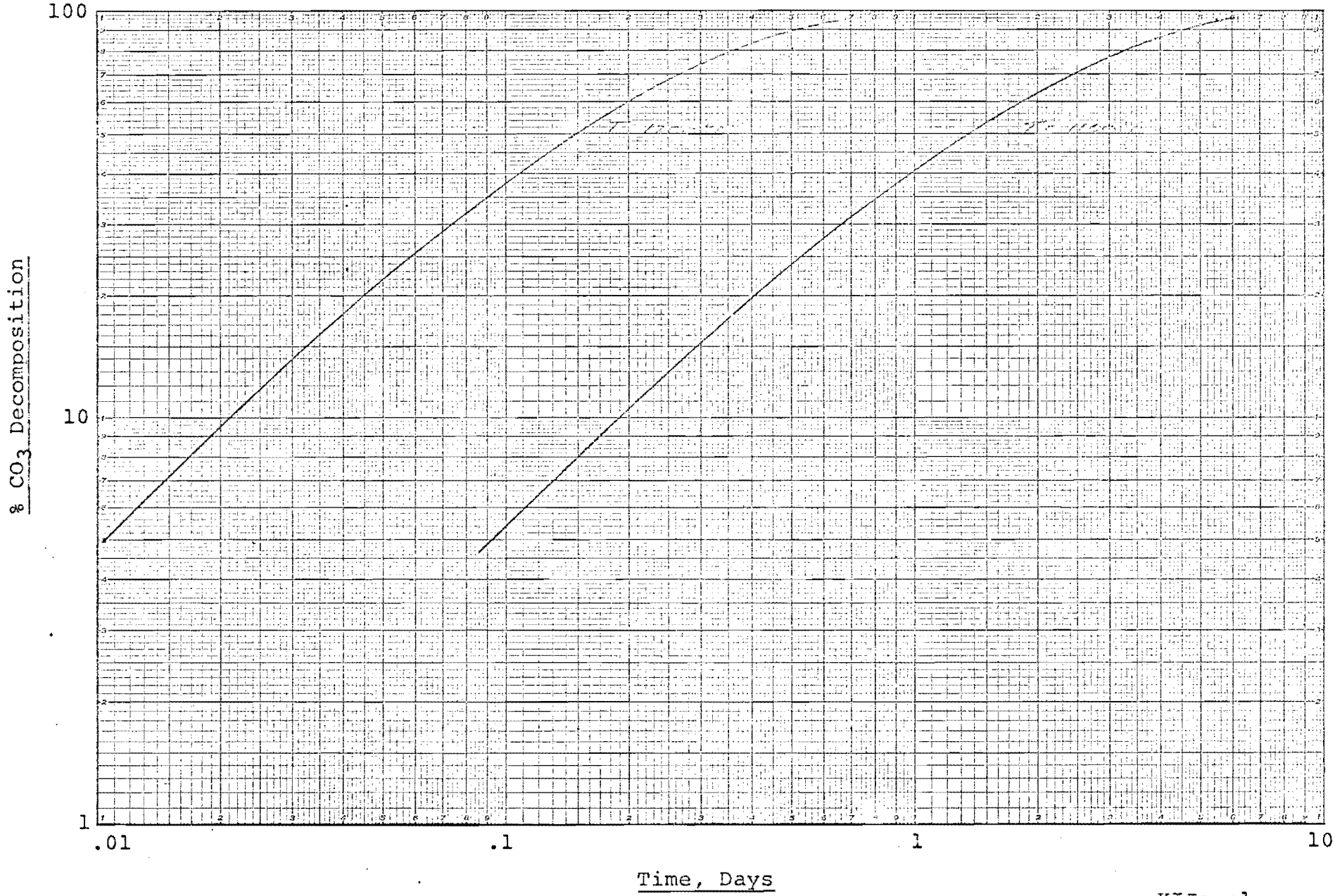
FIGURE 8

CO_3 DECOMPOSITION VERSUS TIME AT 1100 AND 1200 F



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is conceivable that this lower portion of the clinker was in this region near the air distributor manifold for this period of time and was the cause of the observed temperature profile anomalies.

F. Sensitivity of Optimum Maximum Size to Crushing Costs (Revision to December 21, 1966 Monthly Progress Memorandum)

The effect of reasonable variations in crushing costs on the optimum maximum size shale was discussed in Section V, C. of the December 21, 1966 Monthly Progress Memorandum. In reviewing these results, an error was found on Figure 4 of the December 21, 1966 issue. A corrected version of the results of this sensitivity study is shown on Figure 9 of this report.

Analysis of the revised results shows that if incremental size reduction costs more than expected, 2 1/2 inch minus or 3 inch minus shale may well look best. Also, lower incremental size reduction costs will not give an optimum size much below 2 inch minus. (The December 1966 Monthly erroneously showed 2 inch minus as optimum size for all cases.) The amoun of fines produced and processed as well as retorting yields can also affect the optimum maximum shale size. These effects are discussed in the October 21, 1966 and December 21, 1966 Monthly Progress Memoranda.

G. Adjustment to Retort No. 2 Demonstration Run Yields

There has been considerable interest in how Retort No. 2 demonstration run yields were adjusted to a common basis for use in the economics. Table 7 gives the method used in this adjustment. The equation used is basically a regression analysis of the data done before the 1/4 to 1 inch demonstration run data became available. When the 1/4 to 1 inch demonstration run did become available, the regression analysis did not fit it as well as desired, and the equation was only used to obtain yields by adjusting the demonstration run yields.

The commercial operation represents the amounts and sizes of the fractions based on oil shale crushing data from McLanahan. The recycle gas rates were supplied by the Retort Group. Complete descriptions of all the basis can be found in the October 21, 1966 Monthly Progress Memorandum and in the memorandum to be issued on the economics.

H. Retort No. 2 Pressure Drop Correlations

Two errors have been observed in the correlation equation as stated in the November, 1966 Monthly Progress Memorandum for pressure drop above the air distributor. On page 38,

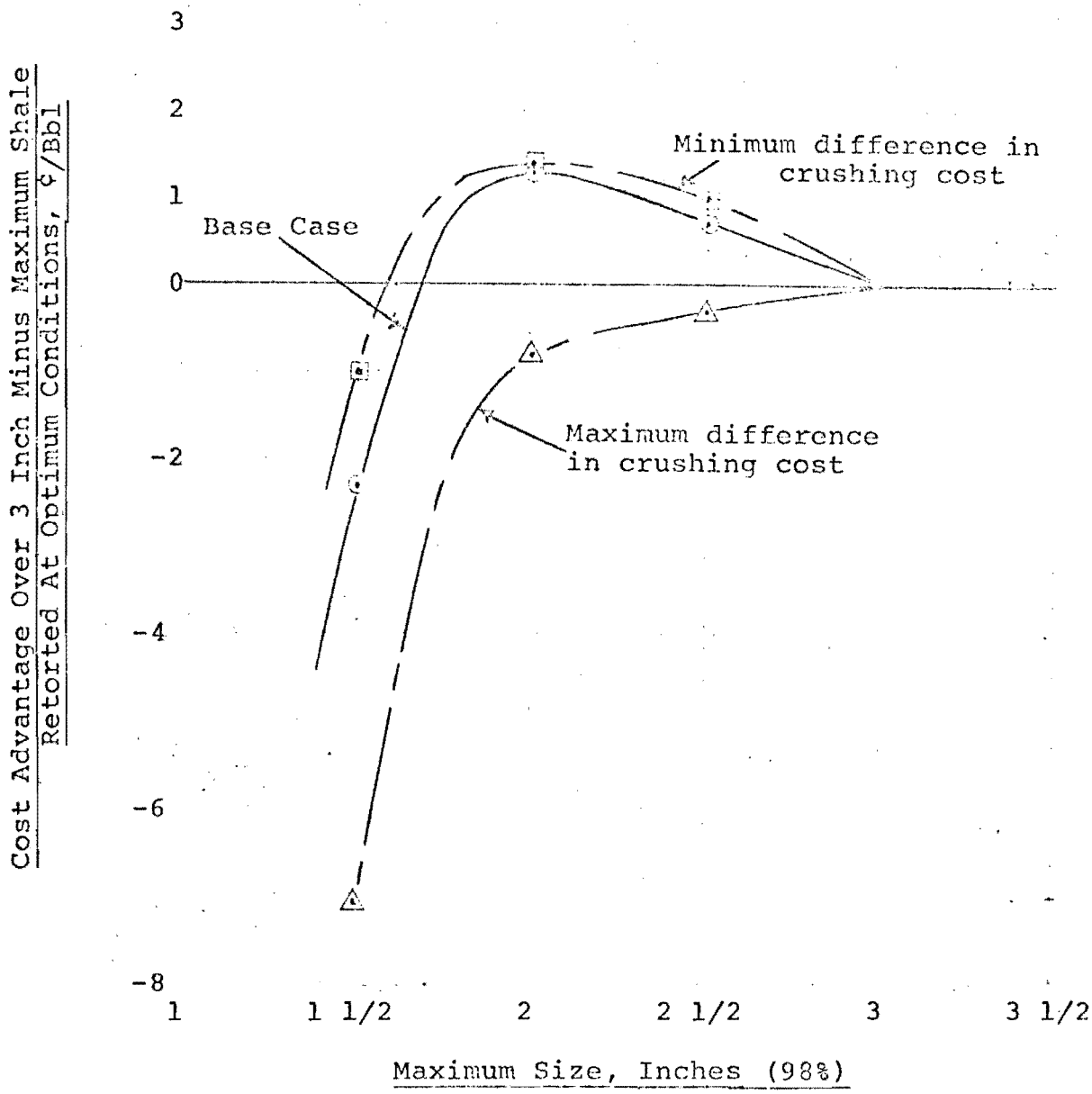
FIGURE 9

EFFECT OF VARIATIONS IN CRUSHING COST ON CHOICE OF MAXIMUM SHALE SIZE

Correction of Figure 4, December 21, 1966 Monthly Progress Memorandum

Base Conditions:

1. Retorting 30 GPT shale to produce 50,000 B/CD of shale oil.
2. Probable crushing cost using a gyratory-cone crushing plant.
3. Retorting one-half the fines at 200 lbs/(Hr)(Ft^2) achieving 85% yield.



RAReitz
1/16/67

TABLE 7

ADJUSTMENT OF DEMONSTRATION YIELDS

	1/4" to 2 1/2"		1/4" to 1"		1" to 2 1/2"	
	953F-N	Comm.	969I-T	Comm.	952F-K	Comm.
Conditions:						
Assay, GPT	27.8	30.0	27.1	30.0	27.5	30.0
Volume Mean Particle Size (Dv), inches	1.36	1.42	.66	.60	1.68	1.75
Area Mean Particle Size (Da), inches	.97	.86	.55	.50	1.50	1.55
Mass Rate, lbs/(Hr)(Ft^2)	492	500	491	500	401	400
Recycle, MSCF/T	14.5	14.3	12.0	12.5	15.1	15.6
Yield Adjustment[1]						
Demonstration Run	86.4		90.8		88.0	
Size Correction, $-1.20(\Delta Dv^2)$	- .20		+ .09		- .29	
Range Correction, $-7.71[\Delta(Dv/D)]$	- 1.92		0.0		- .07	
Recycle Correction, $1.39(\Delta R)$	- .28		+ .70		+ .70	
Mass Rate Correction, $.07(\Delta M)$	+ .01		+ .01		0.0	
Assay Correction, $.36(\Delta A)$	+ .79		+ 1.04		+ .90	
Adjusted Yield[2]	84.8		92.6[3]		89.2	

Notes: [1] Yield adjustments made with the following equation.

$$\Delta yield = -1.20(\Delta Dv^2) - 7.71[\Delta(Dv/Da)] + 1.39(\Delta R) + .07(\Delta M) + 0.36\Delta A$$

A = Raw Shale Fischer Assay, GPT
Da = Surface area mean particle size
Dv = Volume mean particle size
M = Mass rate, 100 lbs/(Hr)(Ft^2)
R = Recycle gas rate, MSCF/T

[2] The adjusted yields may be slightly different than yields used in the economic calculations because all the adjusted yields were plotted and smoothed to give final yields.

[3] The original calculation is 91.6% RSFA. If 92.6% were to be used in the calculations, all of the fractionated systems would show a slight increase in advantage over a one fraction system.

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the coefficient before A should be 0.0641 rather than 0.641 and in Figure 19, which precedes page 39, the constant term should be - 19.324. In summary then,

$$\ln \Delta P_T = -19.324 + 3.4881 \ln T - 1.5041 \ln M - 1.894 Da + 1.050 Dv + 0.0641 A$$

where:
- P_T = Pressure drop, inches H_2O per 10 feet.
- T = Total gas rate, SCF/(Hr)(Ft^2).
- M = Raw shale mass rate, lbs/(Hr)(Ft^2).
- Da = Surface mean particle diameter, inches.
- Dv = Volume mean particle diameter, inches.
- A = Raw shale Fischer Assay, GAL/T.

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

A. Evaluation of Foxboro Total Hydrocarbon Analyzer

The Analyzer determines total hydrocarbons in a gas stream by burning the gas, and measuring the ionization produced by organic carbon present in the gas.

The evaluation of the instrument (which was obtained on a consignment basis) is nearly complete. It was first evaluated by checking its stability at the base line. Then it was further checked by running a standard hydrocarbon mixture for many hours. It was very stable in both cases.

Then, after setting it up with the standard gas, it was used to determine the hydrocarbon content of Retort No. 3 gas for several runs. A comparison of the hydrocarbon-carbon determined by the Analyzer and by our standard laboratory methods was shown in the previous Monthly Progress Memorandum.

The Analyzer gave answers approximately 10% lower than the lab. This is not an unreasonable deviation and could be accounted for by slightly different flow characteristics of the retort and standard gases. A final experiment is planned to verify whether or not this is so.

Although, the instrument is well designed and appears capable of determining the total hydrocarbon in the gas stream, we will probably not buy it for several reasons -

1. It is not likely to give us any information that is vital to the operation of our process.

2. Its cost of about $4,000 is impossible to justify for the relatively short time the project is anticipated to run.

3. We already get the total hydrocarbon content (although admittedly on a composite gas sample) by our normal laboratory analyses.

Therefore, we shall finish our evaluation and then ship it back to Foxboro before the expiration of our consignment period which is February 1, 1967.

B. Quality Control Program Results

1. As a part of our continuing program to keep our laboratory analyses under control, we retain internal control samples which are analyzed periodically by all our technicians.

Recent data have been collected and are presented in Table 8, where they are compared to data obtained about August of 1965. Inspection of the data shows that some problem may exist in the determination of Mineral CO_2 and C-H for raw shale, and for Mineral CO_2 for spent shale. Since we have several new technicians, the decrease in precision on these tests, which require exceptional skills, is not too surprising. We are working now on improving these skills.

All other current precision figures are the same or better than the previous ones.

2. At the same time, we have been doing Fischer Assay analyses of raw shale quality control samples that have been stored under nitrogen since August 2, 1966. The data obtained for the nitrogen-covered samples and those that we normally store in polyethylene bags that are permeable to air are given in Table 9.

TABLE 9

COMPARISON OF FISCHER ASSAY ON RAW SHALE EXPOSED TO NITROGEN OR AIR

	Sample Exposed to	
	Air	Nitrogen
Mean	28.04	28.09
Standard Deviation	0.356	0.335
No. of Samples	32	9

Statistical analysis by the "t" and "F" tests shows no difference either between the averages of the material stored in air or nitrogen, nor between the precision of the data. The experiment will be continued to the end of the program.

C. Liquid Product-Water Analyses for Corrosion Studies

Three samples of liquid product water have been sent to Kilpatrick Associates, Inc. for analysis for pH, Cl^-, $S^=$, Fe, Phenol, and NH_4^+.

Preliminary data have been sent which show pH between 8.4 and 9.0; Fe about 0.5mg/l; and NH_4^+ reported as NH_3, about 1.4%. We will submit three additional samples, which will require some preliminary treatment to prevent losses of the desired constituents. We will report the results as soon as they are available.

TABLE 8

STATISTICAL DATA FROM LABORATORY INTERNAL QUALITY CONTROL PROGRAM

Analysis	Approximate Level	Comparison of Precisions 8/65 SD	8/65 Var.	8/65 DF	1/67 SD	1/67 Var.	1/67 DF	5% F-Ratio Found	5% F-Ratio Critical	Present Results Are -	95% CL 8/65	95% CL 1/67
Raw Shale												
Fischer Assay	28 GAL/T	0.25	0.06029	15	0.36	0.1266	31	2.10	2.25	Same	0.5	0.7
Mineral CO_2	17 wt %	0.10	0.00981	14	0.19	0.0376	19	3.83	2.38	Worse	0.2	0.4
Ash	69 wt %	0.24	0.05638	14	0.09	0.00734	21	7.68	2.41	Better	0.5	0.2
Carbon	16 wt %	0.05	0.00257	16	0.14	0.0201	19	7.82	2.28	Worse	0.1	0.3
Hydrogen	1.6 wt %	0.02	0.000415	16	0.05	0.00284	18	7.04	2.27	Worse	0.04	0.1
Spent Shale												
Mineral CO_2	14 wt %	0.10	0.00989	13	0.17	0.02904	21	2.94	2.46	Worse	0.2	0.4
Ash	83 wt %	0.14	0.01912	18	0.02	0.000308	19	6.2	2.18	Better	0.3	0.0
Carbon	6 wt %	0.06	0.00392	11	0.04	0.001920	19	2.04	2.65	Same	0.13	0.0
Hydrogen	0.2 wt %	0.03	0.001045	11	0.02	0.000308	19	3.35	2.65	Better	0.07	0.0
Gas												
Carbon	9.5 lbs/MSCF	0.21	0.04448	27	0.26	0.06598	5	1.48	2.57	Same	0.4	0.7
Hydrogen	0.3 lbs/MSCF	0.04	0.001257	25	0.02	0.00025	4	5.04	2.76	Better	0.08	0.0

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D. Dust Counting

Because of delays in supplier deliveries of distillation and filtration equipment, no work on dust counting was done last month.

E. Ammonia and Hydrogen Sulfide Contents in Retort Gas To The Laboratory

Because of the interest in determining the ammonia and hydrogen sulfide levels in Retort No. 3 gas lines, several determinations were made on the gas to the laboratory gas holders. A scrubbing train, followed by a wet test meter, was employed. The gas was passed successively through three fitted glass filter gas-washing bottles, the first containing 0.1 N hydrochloric acid and the other two containing 1 M cadmium chloride in 0.1 N hydrochloric acid solution. During the preliminary experiments, the first cadmium chloride scrubber rapidly plugged at the beginning of the experiment. The cadmium chloride fritted disk scrubbers were replaced with two Fischer-Milligan scrubbers containing the same solutions. The advantage of the Fischer-Milligan scrubber is that it does not plug easily. The gas bubbles are fairly large, and the bubbles travel a corkscrew path from the bottom to the top of the scrubber, before they are discharged. The scrubber's main disadvantage is that the individual bubbles are very large and therefore it is possible that poor contact is made between the reacting cadmium chloride solution and the gaseous hydrogen sulfide in the gas bubble.

Retort gas to the lab annex and gas holders was sampled just before the pressure reducing valves. The results were similar in both cases. There was more hydrogen sulfide than ammonia; the results are presented in Table 10.

TABLE 10

AMMONIA AND HYDROGEN SULFIDE CONTENT IN LABORATORY ANNEX RETORT GAS

Run No.	Date(s)	Volume % NH_3	Volume % H_2S
C-990-9	12/29/66		
C-990-18	1/3/67	0.063	0.28
C-990-24	1/6/67	0.034	0.23

F. Shale Oil Disposal - Compatability of Shale Oil With No. 6 Fuel Oil

Shale oil disposal may become a serious problem early in the spring because of limited storage capacity. Although many of the local communities would like to use shale oil for road stabilization, no one of them will take the oil during the winter. The only source of disposal located to date during the winter is Public Service of Colorado. In order to assure Public Service that our shale oil was compatable with their No. 6 fuel oil, a very rough attempt was made to measure the compatability of these two oils at 170 F. Mixtures of 10, 30, 50, 70, and 90% shale oil (remainder No. 6 fuel oil) were heated to 170 F in an oven for 24 hours, and then 48 hours.

Qualitative examination of the oils after 24 and 48 hours showed no pronounced differences. No significant amounts of sludge or sediment were seen, but all observations must be tempered with the knowledge that the solutions were completely opaque to visual viewing.

G. Raw Shale Moisture

In order to check the time required to dry 1 to 2 1/2 inch raw shale, a series of raw shale Ty-lab samples were dried in an oven at 255 F for a period of days. Periodically, the samples were taken out of the oven, weighed, and then replaced. The preliminary results indicate that a 48-hour drying period is sufficient, and that the moisture content of the raw shale is 1.4 to 1.7% by weight.