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NOTICE



SOCONY MOBIL OIL COMPANY, INC.

RESEARCH DEPARTMENT

# MONTHLY PROGRESS MEMORANDUM
(Covering March 16 to April 9, 1965)

ANVIL POINTS OIL SHALE RESEARCH CENTER

Rifle, Colorado

April 23, 1965

CONTRIBUTORS:

Mechanical Engineering
Retorting Section
Analytical Laboratory Section
Engineering and Economic Analyses

Signed by:
RH Cramer
R. H. Cramer
Program Manager

NOTICE

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

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

(1) Socony Mobil Oil Company, Inc., Project Manager

Humble Oil and Refining Company

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

MONTHLY PROGRESS MEMORANDUM

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MONTHLY PROGRESS MEMORANDUM

(Covering March 16 to April 9, 1965)

## I. ADMINISTRATIVE

Technical Observers, representing all of the Participating Parties, have been invited to visit the Research Center during the week of April 12, 1965. This Progress Memorandum was prepared one week early in order to assist them. In the future, it is hoped that Technical Advisory Committee meetings, and consequently Technical Observers visits, will be timed such that the Progress Memoranda can be issued on the regular date.

Two Socony Mobil Retort Engineers, J. G. Mitchell and J. H. Hadded will report about April 12 for a three month assignment.

## II. MINING

### A. Oil Shale Production Mining

Rehabilitation of the Anvil Points mine to provide about 15,000 tons of oil shale for the remainder of Stage I is now about 80 percent complete. The mining plan was approved by the Contracting Officer for the Secretary of Interior on March 26, 1965. A diagram of the mine showing the location of rounds to be blasted, the new power cable and the new underground transformer substation is shown in Figure 1.

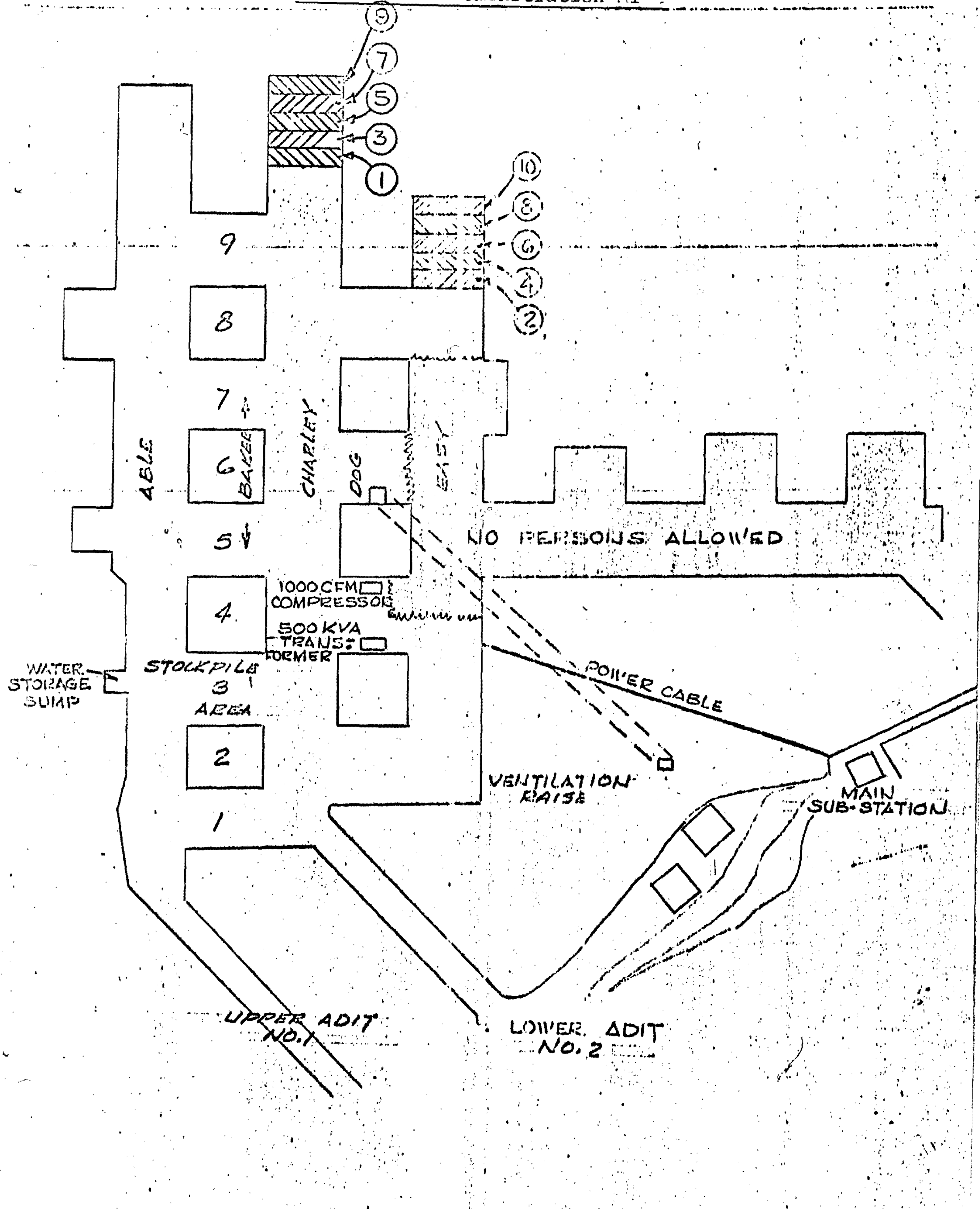
The first round was blasted on April 1 and broke out cleanly. This round had been drilled by the Bureau of Mines prior to shutdown in 1956. The second round is scheduled to be blasted April 21, after rehabilitation is complete.

### B. Joy Rotary Percussion Drilling Demonstration

During the latter part of March a series of drilling tests was carried out in the mine. The tests were carried out by the Joy Manufacturing Company and were observed by representatives from Socony Mobil, Continental Oil, Joy Manufacturing, and Climax Molybdenum Companies. The Joy Rotary-Percussion Drill was used throughout the tests. The drill was mounted on a "demonstration" rig. This rig (having been designed to comply to highway rather than underground mining specifications) was very limited in its provisions for varying and recording the several variables that are associated with rotary-percussion drilling. Furthermore, the rig appeared to be in poor repair when it was introduced into the mine. In view of the above, the information that was obtained during the test period is not as accurate or as detailed as originally planned. However, certain relationships were indicated, particularly during the last three days of the testing

Figure 1

A[illegible]l Points Demonstration Mi



period when the rig seemed to operate free of most of the difficulties that were present during the first two weeks of the testing period.

Average drilling rates of higher order than previously accepted, and possibily as high as 8-10 ft/min, were indicated by these tests. The drilling rate is affected by the oil content of the shale, the higher rates associated with the higher oil content. The drilling rates at right angles or at 45° to the bedding plane of the oil shale appear to be lower than those obtained when drilling parallel to the bedding plane. Drilling dry is essentially as fast as drilling wet. The rate of drilling 2" diameter holes was higher than the rate of drilling 1 5/8" holes.

The rotary-percussion drill utilizes air pressure at 100 psi to create the percussive effort and to provide the feed to the drill. The amount of air required is about 350 cfm. The rotational effort is provided by a hydraulic motor which is activated by a 20-hp hydraulic pump. The water consumption of the drill was estimated at about 1.3 gal/11 ft hole, or 1.12 gal/ft of hole. The average life of bits was estimated at about 3000 ft, (including nine resharpenings per bit). The cost of the bits tested was $14.00 each, when bought in quantity. Over 2300 feet of hole were drilled during the tests, and the rig operators claimed that considerably more footage was drilled in another oil shale property prior to these tests. The same steel rod was used throughout both these testing periods. This indicates that the life of the steel rods may be expected to be over 5000 feet.

In drilling a 20' x 60' (partial) round, over 50 percent of the time was spent in actual drilling, about 10 percent of the time was spent in retracting the steel from the hole, about 33 percent of the time was spent in moving the boom from one hole to the next and collaring the holes, and about 5 percent of the time was spent in delays. On the basis of the above, it is estimated that one man operating one rig which incorporates two rotary-percussion drills and operates at about 80 percent efficiency might be expected to drill a full round (39' x 60') in about three hours.

A Technical Memorandum will be issued covering the results of the Joy test in more detail.

At this point it is recommended additional drilling tests be run with a plain rotary drill. If the plain rotary does not prove as good as the rotary-percussion drill, then additional tests with the rotary-percussion machine should be undertaken.

## III. MECHANICAL ENGINEERING (W. S. Bergen)

Emphasis on Retort No. 1 has centered on revisions necessary to improve carbon balances. Redesign and reconstruction of Retort No. 2 is also progressing with a projected completion date of April 23, 1965.

## Summary

### Retort No. 1

Principal revisions and additions to Retort No. 1 include a new gas sampling system, installation of a time printer for the Richardson batch dumping system, incorporation of a digital voltmeter to set the rate of shale feed, fabrication and installation of the air cooled condenser in the product recovery train, design and installation of a purge gas treating system to provide a gas blanket at the top of the retort, and installation of the product tank mixer.

### Retort No. 2

The new superstructure for the top of the retort was installed and bricked. Design of the change in the spent shale conveyor to accommodate a double star feeder, the water decanter, the air/gas distributor, liquid piping changes, and a purge gas treating system were completed.

### Mechanical Models

Tests of the air/gas distributor designed to the criteria reported in last month's report indicate that air penetration figures must be used on the conservative side of predicted ranges. A new configuration for the Retort No. 2 distributor was designed. One distributor will be used for the 1/4 to 3/4-inch and 3/4 to 1 1/2-inch shale and a different arrangement for 1/4 to 3 and 1 1/2 to 3-inch shale.

## Discussion

### Retort No. 1

Shale rate is now being established using a digital voltmeter. The Taylor rate controller is an electronically actuated unit. The 0 - 10,000 pound/hour rate range is measured over a four inch scale and therefore an exact shale rate can not be set with precision. A digital voltmeter was purchased and installed which measures the set point voltage to the nearest .0001 volts. On this scale, set points may be established to the nearest pound per hour. Subsequent tests showed the Syntron system to be controlling shale rates to ± 1% for any set point for extended time intervals. Repeatability is also in the ± 1% range. This is within stated guarantees.

A Simplex time recorder was purchased and installed. This unit prints the time of each dump of the Richardson system. Further wiring changes are being incorporated to actuate this unit through a completely isolated circuit.

A Lightnin' mixer for liquid product tank T-3 was purchased and installed. This mixer operates at low speed and has a dual bladed stirrer.

In a continuing effort to contain the dust of the spent shale, a cover was installed on the underside of the spent shale conveyor. A dust catch basin was also installed near the head pulley to accumulate dust at the beginning of the belt return. During the last operating period, spent shale dust accumulation was measured at various places. These data will be reported in next month's memorandum.

Smoldering of shale has occurred in the spent shale bin. A nitrogen purge system was installed to blanket the lower section of this bin.

A magnetic head pulley for conveyor "A" was installed. This will eliminate tramp iron from the raw shale feed system.

A new air cooled condenser was fabricated and installed. This unit was tested in Runs 547, 549, 550, and 551. The electrostatic precipitator is not used when the condenser is in operation. Run 547 duplicates Runs 544, 545, and 546 in operating conditions when the electrostatic precipitator was in service. Total product recovery was not affected by the changeover from the electrostatic precipitator to the air cooled condenser. Additional comparative runs are needed, however, to firm this conclusion and evaluate product and gas properties. Runs 549, 550, and 551 were tests of mist recovery system changes. This will be evaluated in next month's memorandum.

Inspection of the electrostatic precipitator during the March 19 shutdown showed no serious deposit buildup on the tube walls.

The retort shell was inspected on the March 19 shutdown and showed no further warping.

The water decanter discharge line diameter was increased to accommodate surges due to pumping the electrostatic precipitator hourly into this vessel.

In an effort to improve carbon balance data, an intensive development program centered around air intake at the top of the retort and sampling procedures. Air was being drawn into the retort due to operating the top retort pressure slightly negative. Air velocities in the shale intake and approximate air drawin rates were developed at different negative pressures. These rates are affected by the top star feeder seal effectiveness at any time. Based on these data, a purge gas system was designed. This system provides sufficient gas to displace the air being drawn into the system. Approximately 10 SCFM is needed for this purpose. To minimize water condensation in the exposed shale feed leg, a water cooled purge gas condenser was designed and installed to cool gas to 70° F. Since this gas is the same as the sampled gas for analytical, this larger volume also provides a better means of evaluating condensables which might go undetected in small gas samples. An isokinetic sample port for 10 SCFM was designed and installed in the total gas piping. Gas flow at this point is constant and unaffected by process variations of shale rate or recycle rate. Gas from the isokinetic port is cooled to 70° F, passes through two glass wool mist eliminators, is reheated to above 100° F, and metered to the purge inlet in the shale feed leg. A side stream for Analytical is obtained just ahead of the purge gas rotameter. Oil and water condensed or separated in this system is collected in two accumulators. This system appears to be effective in treating purge gas, in providing adequate volumes of gas to generate ample water and light hydrocarbon condensate to evaluate the vent gas stream, and in providing a proper sample for analytical purposes. This system will be incorporated in Retort No. 2.

Retort No. 1 will be thoroughly overhauled on the shutdown scheduled for April 6, 7, 8, and 9. All systems will be put into the best condition possible to permit sustained and reliable test runs.

Retort No. 2

Completion of design and construction of Retort No. 2 has been delayed to accommodate the carbon balance program and additional changes to the retort. Completion of construction is now slated for April 23 with testing and calibration completed by April 30.

The spent shale weigh bin and scale addition was approved. Specifications for a scale were developed and a scale ordered. The bin will require new foundations and reorientation. At high mass rates (700 lbs/(hr)($ft^2$) the bin and scale are sized for a three hour capacity.

Results of testing the three pipe distributor system by the mechanical models group were incorporated in a redesign of the air/gas distributor for Retort No. 2. This test data is reviewed in the mechanical models group report. The basis for the Retort No. 2 distributor is described in the attached letter, (WSB to PHC dated April 1, 1965). Essentially the distributors for 1/4 to 3/4 and 3/4 to 1 1/2-inch shale will be 3-inch pipes with 9.4 inch face to face clearances. The distributors for 1/4 to 3 and 1 1/2 to 3-inch shale will be 3-inch pipe with 22.5 inch face to face clearances. Discharge port spacing on the distributor for the smaller shale sizes will be on 6 inch centers to permit large (5/16 inch plus) port diameters. Discharge ports on the distributor for the larger size shale will be on 3 inch spacings with port diameters of approximately 3/8 inch.

# INTEROFFICE CORRESPONDENCE

RESEARCH DEPARTMENT
Anvil Points Oil Shale Research Center
Rifle, Colorado

April 1, 1965

File 760

R. H. Cramer
Program Manager
Anvil Points

AIR-GAS DISTRIBUTOR DESIGN - RETORT NO. 2

The Mechanical Models Group has completed testing of the protype distributor design previously agreed upon. (See letter of March 4, 1965) This new study indicates that revisions be made to the recommendations concerning spacing of distributor tubes for the 1/4 - 3/4 and 3/4 - 1 1/2-inch shale ranges. Essentially, there was less penetration of gas into the shale bed with these two shale ranges.

During this testing a wide range shale, 1/4 - 3-inch, was also processed as requested.

The attached tables show the results of both the two and three pipe distributor studies.

After a careful review of this data, the following distributor configuration was proposed and will be incorporated into Retort No. 2. This proposal was reviewed in detail in a meeting with R. H. Cramer, T. C. Lyons, L. J. Skowronek, and W. S. Bergen.

1. For 1/4 - 3/4 and 3/4 - 1 1/2- inch shale, distributor tubes will have a 9.4 inch clearance. Distributor ports will be spaced on 6-inch centers directed 30° below the horizontal.

   The 9.4 inch distributor clearance is governed by the existing retort width and distributor pipe size. It does represent a judgment of the approximate maximum spacing for these shale ranges. This condition satisfies the 3/4 - 1 1/2-inch shale requirement and slightly exceeds the indicated maximum spacing for 1/4 - 3/4-inch shale. However, effects of wide distributor spacing will be evaluated in service.

   Distributor port center lines were increased from 3-inches to 6-inches to permit the design of ports at least 5/16-inch in diameter and with high differential pressures. (Approximately 16-inches of $H_2O$ at 700 shale mass flow and 6000 SCF air/ton of shale.)

2. For the 1/4 - 3 and 1 1/2 - 3-inch shale, distributor tubes will have 22.5 inch clearances. Distributor ports will be spaced on 3-inch centers directed 30° below the horizontal.

   The 22.5 inch distributor clearance is governed by the existing retort width and distributor pipe size. It is close to the maximum spacing indicated for 1 1/2 - 3-inch shale and slightly exceeds the indicated maximum distributor spacing for 1/4 - 3-inch shale. This spacing was chosen in an attempt to test maximum distributor spacing without jeopardizing retorting efficiency. Effects will be evaluated in service.

Tests will be made of these distributors to check uniformity of air distribution from the ports. Distribution tests will be made to determine the effect of a heated distributor pipe and of heating of the air along the distributor from the inlet to the last port.

These distributors will be limited in their application to shale mass flows between 500 and 700 lbs/(hr)($ft^2$) and air rates at 5000 to 6000 SCF/ton of shale. Tests using preheat or dilution gas will require different distributor ports.

It was the judgment of the group that distributors designed to the above criteria would offer no serious scale-up problems.

W S Bergen

W. S. Bergen

WSB:rl
Attachment

cc: J. E. Lawson
T. C. Lyons
L. J. Skowronek

The purge gas system and condenser has been designed for a 40 SCFM purge rate. This gas will be used to purge the top and bottom feeders. A condenser will be constructed as soon as drafting is complete.

A double feeder is being incorporated for the discharge of spent shale. This will allow balancing the purge against the lower retort pressure. Revisions to the first spent shale conveyor are being made to accommodate this double seal. Provision is also being made to retain and collect spent shale dust for weighing purposes.

The top raw shale feed chute is being completely revised. Two feed chutes with a four foot height adjustment have been designed and are being drafted. One chute will be used for the 1/4 to 3/4 and 3/4 to 1 1/2-inch shale for bed heights of 5 to 9 feet. This will match the shale size - distributor configuration - bed height relationships developed to date. The other chute will be used for 1/4 to 3 and 1 1/2 to 3-inch shale and have a 10 to 14 feet bed height adjustment. It will be necessary to suspend retort operations during raw shale chute and distributor changes.

A water decanter was designed and constructed but will be eliminated from the system in accord with changes found necessary on Retort No. 1. Water will be decanted from the main product tank.

A Lightnin' mixer will be incorporated on the main product tank to thoroughly mix the liquid product.

The air cooled condenser is being fabricated in Grand Junction and will be installed when it arrives at the Research Center.

## IV. RETORTING SECTION (J. E. Lawson)

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

#### 1. Air Distributor Studies

The previous work in this area was concerned with scouting the variables involved in air distribution and was carried out with a 2-pipe model. These studies revealed that shale size was the most important factor governing the distribution.

Recently, the model was modified to handle a 3-pipe section. Thus it was possible to simulate the performance of a full distributor flanked by a half-distributor on either side. This increased the model area which was served by the air distributors and lessened any possibility that the simulated recycle gas could bypass the distributors. In this study, the injection velocity was held constant at 100 ft/sec and the injection ports were angled 30° below the horizontal. The center distributor contained 8 injection ports (4 on each side) and the side distributors contained 4 ports each. Smoke was used to trace the flow patterns as in the past.

The observations of the 3-pipe model are summarized in Table 1. The results of the earlier work are included in the table for comparison purposes. It appears that the penetration is at the lower end of the range that was indicated earlier. Observations were also made with the full-range, 1/4 to 3-inch shale which had not been included in the scouting studies. The penetration of the combustion air into this material is in line with the other shale sizes.

The overall study was carried one step further in an attempt to determine if the depth of penetration is greater in a moving bed. In order to do this the depth of shale above the distributor pipe was increased from the standard 12 inches to 21 inches. After the pattern was established for the static condition, the bed was lowered while movies were taken in slow motion. An analysis of the movies indicated that there was a momentary surge in the pattern as the bed loosened. However, as the entire bed began to move the pattern returned to its original shape rather quickly. As the shale level passed the standard height of 12 inches there was no significant difference from the pattern observed in the static bed. This was the case for both the 1/4 to 3/4 and 3/4 to 1 1/2-inch shale sizes (the size of the shale drain on the model limited the drawdown studies to these smaller sizes). Thus, it is concluded that no additional credit in penetration should be taken for the fact that the bed is moving.

As a result of the somewhat lower penetrations indicated by this recent work, steps have been taken to make further studies of the minimum clearances required for continuous flow of the larger (3 inch) shale sizes. It is known that the minimum clearances in Table 1 for the 1/4 to 3-inch and 1 1/2 to 3-inch sizes are conservative because of physical limitations of the shale flow model. (Slot sizes approached a square rather than rectangular as in the smaller shale sizes.) If these minimum clearances could be determined more accurately, a tighter design could possibly be incorporated in the distributor for Retort No. 2 and this could improve the chances of obtaining a single design that would be satisfactory for all shale sizes.

The shale flow model has been modified and these studies are in progress.

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

### A. Carbon Balance Problem

Much of the laboratory's effort during the past few weeks has been directed toward investigating the carbon balance problem.

Table 1

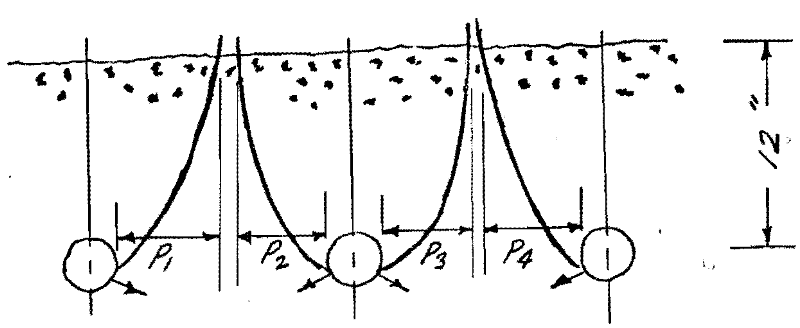
SUMMARY OF AIR DISTRIBUTOR STUDY
TWO DISTRIBUTOR SYSTEM

| Shale Size (in) | Minimum Clearance Required For Shale Flow (in) | Air Penetration From A Single Distributor (in) | Air Penetration From Two Opposing Distributors (in) | Maximum Port Spacing On Distributor (in) |
|---|---|---|---|---|
| 1/4-3/4 | 2 | 3 1/2 - 5 1/2 | 7 - 11 | 6 |
| 3/4-1 1/2 | 4 | 5 1/2 - 7 1/2 | 11 - 15 | 9 |
| 1 1/2-3 | 14 | >10 1/2 | >21 | 12 |

SUMMARY OF AIR DISTRIBUTOR STUDY
THREE DISTRIBUTOR SYSTEM

| Shale Size (in) | Minimum Clearance Required For Shale Flow (in) | Air Penetration[(1)] From Each Distributor | | | | Average Penetration (in) | Average Air Penetration From Two Opposing Distributor (in) |
|---|---|---|---|---|---|---|---|
| | | $P_1$ (in) | $P_2$ (in) | $P_3$ (in) | $P_4$ (in) | | |
| 1/4-3/4 | 2 | 3 1/2 | 4 | 4 3/4 | 3 1/2 | 4 | 8 |
| 3/4- 1 1/2 | 4 | 4 3/4 | 5 3/4 | 6 | 4 1/2 | 5 1/4 | 10 1/2 |
| 1 1/2-3 | 14 | -- | 12 1/4 | 12 3/4 | -- | 12 1/2 | 25 |
| 1/4-3 | 10 | -- | 9 1/4 | 9 1/4 | -- | 9 1/4 | 18 1/2 |

(1)See Sketch below



The results of this work are included in another section of this memorandum.

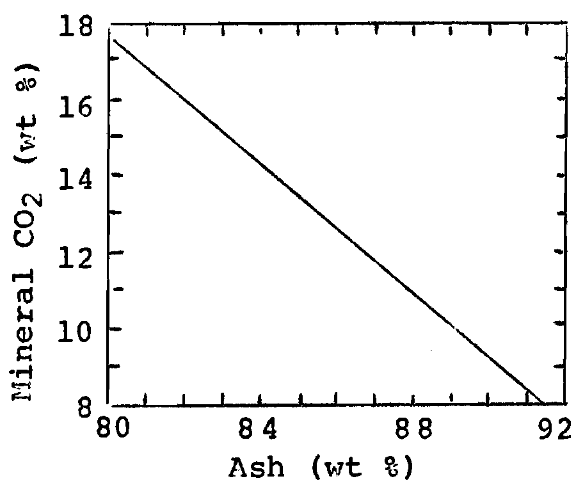
B. Relationship for Spent Shale Analyses

In last month's Progress Memorandum several relationships among analyses on raw and spent shale were reported. Since that time another one has been found that has proven useful in checking our results.

For spent shales having Fischer Assays of 0.0 gal/ton, the following relationship has been determined by regression analysis.

Mineral $CO_2$ vs Ash

Runs 465 through 509 (N=37)
$CO_2 = -0.859(\text{Ash}) + 86.44$
$R = 0.97$
$s = 0.32$




C. Nitrogen in Crude Shale Oil

The consistency of the nitrogen content of crude shale oil has been observed for several months. As an example, the oils from 66 recent runs (Nos. 465 through 536) showed an average nitrogen content of 2.32 wt % with a standard deviation of 0.067. These oils were retorted from shale ranging from 24 to 33 gal/ton. In view of this consistency and the use which is made of this determination, effective with Run No. 538. nitrogen will no longer be regularly determined on crude shale oil.

D. Quick Assay of Spent Shale

The retort section requested the laboratory to develop a rapid test to determine the amount of potential oil left in spent shale. This is routinely determined from the Fischer Assay;

however this test requires several hours. To satisfy this request, a method used by the Bureau of Mines to estimate oil yield of lean raw shale was adapted ["Estimating Oil Yield of Lean Oil Shale", K. E. Stanfield, Anal. Chem. 25, 1552 (1953)].

The procedure involves heating a 3-gram sample of minus 8-mesh shale in a test tube for five minutes. The test tube is situated in a furnace maintained at 600 ± 50° C; approximately the upper third of the test tube extends out of the furnace. When the sample is heated, oil is retorted from the shale and condenses on the cool portion of the test tube. The appearance of the "carbon ring" is related to the Fischer Assay of the shale. Seven standard heated test tubes containing shale from 0.0 to 4.3 gal/ton have been prepared for comparison. Spent shale of unknown oil content is heated according to the procedure, and the resulting appearance of the test tube is compared to the standards to estimate the Fischer Assay of the spent shale.

## VI. PROGRESS ON IMPROVING CARBON BALANCES (Joint effort of the Retort, Analytical and Engineering Analysis Groups)

### A. SUMMARY

#### 1. Accomplishments to Date

A bias in carbon balance with yield was observed with past data (re March Progress Memorandum, page 19). Therefore, a series of about 25 runs have been made on Retort No. 1 where sampling equipment and procedures were carefully reappraised; and changes were made where indicated improvements in accuracy appeared possible. OSRC-11 Summary Sheets covering these runs are attached.

In this extensive program to improve organic carbon balances, a number of changes were investigated and implemented in procedure, equipment, and sampling. These modifications have improved carbon balances and have expanded our understanding of carbon balance problems.

The major improvements in organic carbon balances were developed in two main areas: (1) improved air measurement and accountability and (2) improved carbon measurement on vent gas.

##### a. Air Measurement and Leakage

Accurate air measurement and negligible air leakage into vent gas are essential to good organic carbon balances. Vent gas rate is calculated in our material balances by a nitrogen balance around the retort, assuming the nitrogen exiting in vent gas is equal to the nitrogen coming in with the air. Thus vent gas rate is equal to 0.79 A/Nvg, where A is air rate and Nvg is volumetric nitrogen percentage in the vent gas. Since nitrogen is about 60% in the vent gas, any air measurement error is enlarged to a vent gas error of 1.3 times as much. Since mineral $CO_2$ composition of the vent gas is calculated from the difference in mineral $CO_2$ content of the raw and spent shale, the entire error in total carbon contributed by the vent gas is manifested in terms of organic carbon.

An error in air measurement of 500 SCF/Ton RS represents an error in vent gas rate then of 650 SCF/Ton, or about 8.5 lb Carbon/Ton RS. This represents 3.5% on organic carbon balance. Thus an error in air rate measurement of about 10% results in an organic carbon balance error of 3%.

An air leak into the vent gas affects nitrogen balance and, consequently, organic carbon balance in a similar manner.

The meter for measuring air into the retort has been installed according to standards and is being recalibrated to insure accurate air measurements on future runs. Another section of the report describes the orifice calibration made after Run 529. In the future the air meter orifice will be routinely calibrated and a dry test meter will be used in series with the air orifice.

An air leak into the top of the retort was also found during Run 529 and is discussed in another section. Vent gas is now being used as a seal gas to prevent leakage of air into the raw shale feed pipe. In addition, a second star feeder is to be installed at the top of the retort to insure against air leakage.

b. Improved Carbon Measurement on Vent Gas

Sampling of the vent gas is particularly difficult because of fine mist. In earlier runs, the gas sample was obtained by running a sample line from the retort vent gas line to a gas holder in the laboratory, where it was analyzed for total carbon. The sample line entered the vent gas line in a horizontal run and extended into the line about 1-inch. The sample was not isokinetic and may not have been representative of the vent gas stream.

The vent gas sampling has been improved in two ways - first a larger measured 10 CFM stream, used as a seal gas for the top of the retort, is sampled isokinetically from the total recycle stream, is cooled and the condensate is measured; - second, the uncondensed gas is sampled and analyzed. The larger seal gas stream is believed to be more typical of the vent gas composition.

Mist in the vent gas is now determined from the condensate collected from the condenser. Results to date indicate that more carbon is present in vent gas than was found by the previous sampling method. The condensate represents much more carbon than was found by the jet impactor test used previously. The condensate also appears to vary inversely with yield and partially explains poor carbon accountability at low yields. These sampling improvements will be discussed in detail later.

The improved sampling of vent gas has resulted in an increase in carbon in both the non-condensable gas and the condensable liquid collected. Both have contributed to the noted improvement in organic carbon balances.

The following summary of results from low and high yield operation, before and after these changes illustrate their effect.

| Operation | Yield Vol. % RSFA Tank | Yield Vol. % RSFA Mist & Cond. | Dry Gas SCF/T | Org. Carb. Bal. | Org. Carbon Distr., Wt % Org. Carbon Gas | Org. Carbon Distr., Wt % Org. Carbon Spent Shale | Product Analyses Dry Gas(1) Btu SCF | Product Analyses Spent Shale Total Carbon, Wt % |
|---|---|---|---|---|---|---|---|---|
| Low Yield | | | | | | | | |
| Before | 78 | 0.2 | 6600 | 92 | 19 | 13 | 120 | 6.0 |
| After | 79 | 2.0 | 6800 | 101 | 26 | 16 | 140 | 6.8 |
| High Yield | | | | | | | | |
| Before | 90 | 0.2 | 7300 | 101 | 23 | 12 | 100 | 5.4 |
| After | 88 | 0.7 | 8000 | 102 | 25 | 12 | 110 | 5.0 |

(1) Dry gas excluding mist and condensate

The higher dry gas make after the changes is the result of the higher measured air rate and elimination of the air leak. The higher heating value of the gas is the result of the improved gas sampling location. The higher yield of mist and condensate is the result of cooling a larger gas sample and the isokinetic sampling. The higher carbon content of the spent shale after the change is not the result of any changes in the spent shale system, but due rather to the reduction of oxygen in the recycle gas by eliminating the air leak in at the top of the retort. Operation at low yields now shows the converted oil showing up in the gas and spent shale as products of cracking or combustion. Significant errors in oil measurement were not uncovered and therefore reasonable confidence can be placed in past oil yields.

2. Modifications Still Under Consideration

During this study the following modifications to Retort No. 1 were indicated as potential additional improvements for precision in results and measurement of process conditions.

a. Installation of positive gas seals at the top and bottom of the retort. This will be implemented by installing double star feeder with a pressure balance across the inside feeder. This improvement will increase the precision of the actual recycle rate determination and increase confidence in the gas make calculation.

b. Increasing the spent shale sample size from 1 to 3% and modify the sampler, crusher and splitter to eliminate dust loss from the sample. This will provide a more representative sample and increase the precision in ash balance, organic carbon balance, and mineral $CO_2$ decomposition.

c. Checking the $N_2$ balance method of determining the dry gas production by means of a helium tracer technique on several runs.

d. Pressure testing the suction side of the oil recovery system and the entire air feed system. This will eliminate minor air leaks in or out of the system which may still exist.

e. Considering the purchase and installation of a continuous carbon analyzer on the vent gas sample. This will improve our precision and accuracy in determining the carbon content of mist laden gas.

f. Considering the advisability of increasing run length from the present eight hour balances to two - eight hour balance periods. This would reduce the significance of potential holdup and raw shale quality variations on results and increase reliability by a factor of 1.4.

g. Considering adsorption analyses of the vent gas for unaccounted for heavy hydrocarbons if further low organic carbon balance runs arise.

## B. DISCUSSION

### 1. Summary of Retort Runs for the Carbon Balance Study

Twenty five runs were made in the carbon balance improvement program. A running summary of the results from these runs is included in Tables 2 through 6. A description of these runs is presented briefly below.

#### a. Runs 526 - 529

Runs 526, 527, and 529 were made to duplicate process conditions of Run 476, a low-yield, poor carbon balance run.

No changes in operating procedures had been made at this time. The low yield of Run 476 was essentially duplicated and so, unfortunately, was the low carbon balance. Run 528 was made to duplicate Run 505, another low yield, low carbon balance run, with essentially the same result.

b. Run 530

Just prior to this run, it was observed that air was leaking into the retort at the top star feeder at a rate approximating 500 SCF/T of raw shale. The top pressure was raised slightly in an attempt to prevent this leak, and conditions of Run 476 were again duplicated. An improvement in both carbon balance and yield were obtained, probably because of the variation in actual process conditions attained through partial elimination of the air leak.

c. Runs 531 - 534

Prior to this series of runs, a vent gas purge condenser was installed, and blanketing of the top star feeder with dry vent gas was initiated. Process conditions of Run 476 were again duplicated. In Runs 533 and 534, use of the oil condensate from the vent purge condenser to estimate mist lost in the vent gas was begun. Yields duplicated those of Run 530 and carbon balances showed a slight improvement, particularly those of Runs 533 and 534 where a greater amount of mist in the gas was estimated.

d. Runs 535 - 542

In an effort to demonstrate good carbon balances under low yield conditions, process conditions for these runs were varied by lowering bed height to 6 feet, while retaining other process conditions the same as for Run 476. Yields showed the expected decrease; however, carbon balance for Runs 535 and 536 also deteriorated. At this time, an air meter calibration had been obtained and estimates of process conditions and carbon balances from Run 538 onward reflect this change. The low yields of Runs 535 and 536 continued; however, the change in estimated air input and gas production caused a marked increase in carbon balance.

e. Runs 543 - 547

These runs were undertaken to demonstrate good carbon balances under high yield conditions. Conditions were arranged to duplicate those of Run 515. As expected, yields showed a marked improvement and carbon balances, for the most part, were very satisfactory. In Run 547, a condenser was used in the mist recovery train rather than the electrostatic precipitator with essentially no change in results.

f. Runs 549 - 551

These runs were made to further demonstrate control of carbon balances under high yield conditions. Air rates were lowered from those of Runs 543 - 547, while total gas rates were maintained. The electrostatic precipitator was not in service. Again, carbon balances were satisfactory.

2. Detailed Reappraisal of Method Used to Measure Rates and Composition of Charge and Product Streams

a. Raw Shale

(1) Sample Splitter

In order to be sure that there was no bias in the raw shale sample splitter a sample was carefully riffled from the splitter reject and compared with the normal sample. The nearly identical Fischer Assay shown below, indicates adequate mixing and splitting of the raw shale.

RAW SHALE SPLITTER REJECT VERSUS NORMAL SAMPLE

| | |
|---|---|
| Normal Sample | 31.2 gal/ton |
| Reject Sample | 31.4 gal/ton |

The sample cutter, which catches a sample from the end of the conveyor belt, was also observed in operation and judged to be as representative as could be obtained.

(2) Raw Shale Weighing System

The batch Richardson weigh bin is being used as the most accurate device for determining the raw shale rate. This device weighs 400-pound batches of shale and dumps them on to the Syntron rate controller to feed the conveyor belt. This was tested against the spent shale scale. Ten dumps weighing 4017-pounds on the Richardson were conveyed through the retort, empty to empty, and weighed 4040-pounds on the spent shale scale. This 0.6% difference is the best we can expect.

A dead weight test also checked the reliability of the single dumps as shown below:

DEAD WEIGHT TEST OF RICHARDSON RAW SHALE SCALE

| Date | Weight Added | Scale Reading |
|---|---|---|
| 3-23-65 | 50 | 49.5 |
| | 100 | 99.5 |
| | 150 | 150.0 |
| | 200 | 200.0 |
| | 250 | 250.0 |
| | 300 | 300.5 |
| | 350 | 350.5 |
| | 400 | 400.0 |
| 3-24-65 | 400 | 400.0 |
| 3-29-65 | 400 | 400.0 |
| 3-30-65 | 400 | 399.5 |
| 4-7-65 | 400 | 400.0 |

A practice of dead weight testing the Richardson scale on a daily basis has been initiated along with periodically running a dynamic test against the spent shale.

3. Timing of Raw Shale Sampling

A practice of starting the raw shale sample cutter 45 minutes prior to the beginning of the run and stopping it 45 minutes prior to the end of the run has been initiated because of the average 1 1/2 hour residence time in the retort. This will provide a sample which is more representative of the shale which is actually retorted during the run.

b. Liquid Product

(1) Potential Liquid Holdup

The possibility of variations in oil level in the liquid product gathering system was evaluated. The only spots for possible variation are in

(a) The dust surge drum in the offgas line right after the retort - this was drained hourly and the oil added to the liquid product tank in order to eliminate this possibility during this study. A drain leg will be added to the bottom of this vessel to permit continuous draining during future runs where dusting is not a problem. When dusting is a problem the vessel will be manually drained.

(b) The continuous water decanter just ahead of the liquid product tank is also a source for changes in oil inventory because of plugging or surging of the water-oil interface. It was checked periodically but because of the difficulty in readily determining the location of the interface, it will be taken out of the system. In the future, water will be decanted hourly from the liquid product tank.

(2) Calibration of Liquid Product Scales

The liquid product scales were calibrated with 400 pounds of dead weight on March 4 and, when rechecked with 1400 pounds on March 22, no deviation was found. The retort operators then checked the scale daily by adding 400 pounds of dead weight. Their check showed an average 0.9% understatement of the weight from Run 537 through Run 544, and an average 1.3% understatement of the weight from Run 545 through 551. These calibrations were used in stating the liquid product weight for the material balance and yield calculations. Henceforth, this balance will be readjusted when found beyond ± 0.5% of the dead weight. The scales are calibrated on a routine basis and cleaned periodically.

(3) Liquid Product Sampling

A Lightnin' mixer has been installed to assure complete mixing of the water and thus a more precise water analysis.

A representative sample is obtained from the final liquid product for carbon analysis.

(4) Weathering of Oil From Tank

Possible loss of oil due to weathering was evaluated by both the retort and analytical groups.

A sample of oil from the bottom of the cyclone and from the liquid product tank was taken and held at 100° F and 140° F for a total of 9 1/2 hours in an open vessel. The results shown below indicate less than 1% loss of oil.

TOTAL % WT LOSS

| Time, hours | 110° F Liquid From Bottom of Cyclone | 110° F Liquid From T-3 Tank | 140° F Liquid From Bottom of Cyclone | 140° F Liquid From T-3 Tank |
|---|---|---|---|---|
| 1 | 0.20 | 0.42 | -- | -- |
| 2 | -- | -- | 0.08 | 0.11 |
| 3 1/2 | 0.35 | 0.47 | -- | -- |
| 4 | -- | -- | 0.39 | 0.46 |
| 6 | -- | -- | 0.63 | 0.78 |
| 9 1/2 | 0.40 | 0.76 | -- | -- |

The retort group found the loss even less significant by observing the weight of the liquid product over a six hour period and finding only a one pound loss out of about 1000 pounds of oil.

(5) $CO_2$ Solubility

Samples of oil from the product cyclone were heated and purged with $CO_2$ - free air which was then passed through a $CO_2$ absorbent. The increase in weight of the $CO_2$ absorber is an indication of the maximum $CO_2$ absorbed in the oil. The results shown below show this amount to be very small.

CARBON DIOXIDE IN LIQUID PRODUCT FROM CYCLONE

| Run No. | Purge Time, hours | Total $CO_2$ Collected Wt mg | Total $CO_2$ Collected % Wt |
|---|---|---|---|
| 530 | 2 | 52 | 0.006 |
| | 4 | 84 | 0.010 |
| 531 | 2 | 26 | 0.003 |
| | 4 | 48 | 0.006 |
| | 6 | 53 | 0.006 |

Analyses of a water sample showed 0.97 wt % dissolved $CO_2$; this would amount to only one pound $CO_2$ (0.3 pounds carbon) per ton of raw shale which is also insignificant.

c. Spent Shale

Spent shale measuring, sampling and analytical techniques have been thoroughly evaluated for possible improvements in carbon accountability.

(1) Calibration of Spent Shale Weight Scales

The Toledo spent shale weigh scale was calibrated with the following results:

| Date | Standard Weight Added | Scale Increase | % Error |
|---|---|---|---|
| March 22 | 1400 | 1400 | 0 |
| March 29 | 1000 | 996 | 0.4 |

The scale is checked daily against an added standard weight of 1000 pounds. The scale increase on this calibration is generally 1000 pounds, thereby indicating that the spent shale scale is a reliable measuring device.

(2) Sampling and Analyses of Spent Shale

A number of potential problems are present in sampling and analyzing spent shale. A thoroughly searching program was initiated to ascertain the extent to which some of these potential problems affect organic carbon balances.

(a) Oxidation of Spent Shale and Volatilization of Condensed Hydrocarbon

One potential problem is that the carbon on spent shale may oxidize to $CO_2$ and be lost on the conveyor and in the spent shale bin. In such a case, carbon analyses on the spent shale will be low. A second potential problem is that the spent shale sample will oxidize prior to analyses and, therefore, indicate a low carbon result. This may occur either by oxidation of iron or sulfur compounds, which would thereby decrease the percent carbon or by oxidation of carbon to $CO_2$ which would then be lost. A third potential problem is that condensed hydrocarbon on the spent shale may be volatilized and lost to the air again resulting in poor carbon accountability. A number of experiments were run to test these hypotheses.

i. Laboratory Tests - Experimental Procedure

Samples of spent shale were allowed to pass from a port located between the turntable and star feeder into a bucket containing liquid nitrogen.

The samples were then heated to 500° F in a nitrogen atmosphere, then cooled in nitrogen. A weight change would indicate loss of volatile matter such as condensed oil.

The samples were also heated to 500° F in a nitrogen atmosphere and then allowed to cool in air. This simulated dropping of the hot shale from the star feeder onto the spent shale belt and conveying it to the spent shale hopper. A weight change would indicate oxidation of some of the carbon.

The quenched spent shale was heated to 500° F in air for various lengths of time to simulate conditions that might exist in the spent shale. Loss in weight, carbon, hydrogen, mineral $CO_2$, and ash were determined. Changes in the concentrations of carbon, especially would indicate the possibility of sample oxidation.

A practical control check of sample oxidation was also made by splitting the spent shale into two sample buckets, one blanketed with nitrogen and one open to the air as usual.

ii. Results

(i) No Hydrocarbon Volatilization

Table 2 Section A shows that the spent shale sample quenched in nitrogen and heated to 500° F does not lose weight when allowed to cool to normal temperatures in either air or nitrogen. This indicates that no hydrocarbon is lost in normal cooling and oxidation appears to be insignificant.

(ii) Carbon Loss Due to Oxidation

Samples obtained in a similar manner and heated to 500° F in air show a loss of weight, as shown in Table 2 Section B. These data and those in Table 3 indicate an oxidation and loss of carbon with time from the spent shale.

Spot temperatures at the bottom of the bin have been observed as high as 400° F and storage time in the bin averages six hours for a 12-hour run. Furthermore, indications of oxidation have also been observed. Occasionally, the retort operators have observed, when dumping spent shale at night, that the material at the bottom gate glows. At other times, they have seen a white ashy appearance in the first spent shale removed from the bin. Hence, it can be concluded that oxidation of carbon probably does occur to a measurable extent in the bin. As a result of this work the spent shale bin has had a nitrogen purge system installed to displace oxygen from the bin and reduce carbon oxidation.

## Table 2

### Shale From Between Turntable and Star Feeder Quenched in Liquid $N_2$

A. Volatilization and Oxidation Experiment

(1) Volatilization Experiment (Heat to 500° F in $N_2$ - Cool in $N_2$ Gas

(2) Oxidation From Star Feeder to Belt (Heat to 500° F in $N_2$ - Cool in Air)

| Run No. | Orig. Wt. g | Weight After Treatment: Cooled in $N_2$ | Weight After Treatment: Cooled in Air |
|---|---|---|---|
| 528 | 234.7 | 234.7 | 234.7 |
| 529 | 249.3 | 249.2 | 249.3 |

B. Oxidation at 500° F in Air

| Run No. | Ignition Time, hrs | Shale Wt., g | Wt. Loss, % |
|---|---|---|---|
| | 0 | 67.52 | -- |
| 528 | 2 | 67.45 | 0.10 |
| | 4 | 67.14 | 0.56 |
| 529 | 0 | 89.80 | -- |
| | 4 | 89.40 | 0.45 |
| | 0 | 66.53 | -- |
| 530 | 2 | 65.77 | 1.14 |
| | 4 | 65.76 | 1.16 |

## Table 3

### Comparative Analyses of Spent Shale Samples With Different Treatments Before Analysis

| | % Component | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Run No. | $C_R$* | $C_Q$ | $C_I$ | $H_R$ | $H_Q$ | $H_I$ | $CO_{2R}$ | $CO_{2Q}$ | $CO_{2I}$ | $Ash_R$ | $Ash_Q$ | $Ash_I$ |
| 528 | 5.08 | 5.20 | 3.59 | 0.11 | 0.10 | 0.08 | 11.8 | 9.7 | 8.4 | 87.6 | 88.8 | 91.1 |
| 529 | 5.63 | 5.33 | 4.25 | 0.11 | 0.08 | 0.08 | 12.8 | 11.2 | 14.3 | 86.0 | 87.4 | 87.4 |
| 530 | 5.33 | 5.56 | 3.34 | 0.06 | 0.11 | 0.08 | 12.3 | 12.3 | 10.5 | 86.2 | 86.0 | 89.4 |

### Loss of Carbon With Ignition in Air at 500° F for 4 Hours

| | C, lbs/ton spent shale | | | |
|---|---|---|---|---|
| Run No. | Q* | I | Loss | % Loss of C |
| 528 | 104 | 71 | 33 | 31 |
| 529 | 107 | 85 | 22 | 20 |
| 530 | 111 | 67 | 44 | 40 |

*Subscripts "R", "Q", and "I" indicate the regular sample from the retort, the quenched sample, and the 4-hour ignited sample, respectively.

The spent shale sample is also being taken under nitrogen. However the rapid cooling of the spent sample to ambient temperature may obviate the need for collecting this sample under nitrogen. Samples taken under nitrogen show no significant difference in carbon content from those taken in air as shown in Table 4.

(3) Analysis of Dust

Spent shale from the bottom of the retort contains dust. Some of this dust is lost out the stack, on the floor and some sticks to the belt conveyor and is lost up the dust stack. While this dust is a relatively small loss, it might represent a significant carbon loss if it, for some reason is especially high in organic carbon content. However, Table 5 which show the results of the analyses of the dust did not indicate an exceptionally high carbon content for the spent shale. The ash and total carbon contents are near that of average spent shale.

The data seem to show a small difference between the composition of dust and spent shale. This indicates that spent shale sampling can be a problem. Thus, modifications and improvements to the spent shale crusher is being made to prevent loss of fines.

(4) Spent Shale Sample Size

The spent shale sample size was about 1%. Sampling errors may be introduced when a solids sample is too small. Bureau of Mines information indicates that a 3% sample should be obtained for it to be representative. Therefore our sampling procedure has been changed to obtain a 3% sample of spent shale. The spent shale sample is also being time-sequenced 45 minutes after the beginning and end of the run, in order to obtain the sample which was actually retorted during the run.

(5) Spent Shale Crushing and Splitting

The possibility was investigated of some selective classification of the spent shale sample during crushing and splitting. Dust losses during crushing of the samples in the retort area are selective losses which could affect analyses of the spent shale.

Experiments were run to determine whether sampling errors did occur in crushing and splitting. All of

## Table 4

Comparison of Spent Shale Analyses on Samples Taken With and Without a Nitrogen Blanket

| Run No. | With $N_2$ Blanket %C | %H | $CO_2$ | Ash | Without $N_2$ Blanket %C | %H | $CO_2$ | Ash |
|---|---|---|---|---|---|---|---|---|
| 531 | 5.82 | 0.12 | 13.6 | 85.0 | 5.86 | 0.10 | 13.1 | 85.4 |
| 538 | 5.50 | 0.15 | 13.0 | 85.6 | 5.48 | 0.16 | 12.8 | 85.7 |
| 539 | 6.43 | 0.16 | 15.8 | 82.4 | 6.53 | 0.16 | 15.3 | 82.5 |
| 540 | 6.36 | 0.12 | 16.3 | 82.1 | 7.22 | 0.16 | 16.1 | 81.7 |
| 533 | 5.96 | 0.14 | -- | -- | 6.05 | 0.19 | -- | -- |
| 534 | 5.87 | 0.13 | -- | -- | 6.16 | 0.12 | -- | -- |
| 535 | 5.58 | 0.11 | -- | -- | 5.50 | 0.14 | -- | -- |

## Table 5

### Analyses of Dust From Retort Area

| | C | H | Ash | Min. $CO_2$ |
|---|---|---|---|---|
| Under Spent Shale Belt 3/10/65 | 5.77 | 0.17 | 89.6 | 7.9 |
| Inside Hopper Shed | -- | -- | 87.5 | -- |
| Inside Casing of Belt | 5.67 | 0.21 | 88.2 | 8.4 |
| Typical Spent Shale For Same Time | 5.8 | 0.14 | 85 | 13 |

the 120-pound sample of spent shale was sent to the laboratory where 2 to 3-pounds were split out, ground and riffled. The remainder of the sample was returned to the retort where it was crushed and split in the usual way to give another sample. Both samples were analyzed and the results are shown in Table 6.

The results are rather inconclusive. The laboratory crushed and the retort crushed samples appear to check reasonably well with one another. However, the small analytical differences do indicate some dissimilarity in the samples. Small deviation in ash analyses can cause larger differences in organic carbon balance when material balances are done on an ash - spent shale bases. In this case, the spent shale ash analysis determines the raw shale rate and hence the amount of ingoing carbon. The carbon content of the spent shale will cause an error in the same direction. For example, on Run 550 use of the analytical-crushed results versus retort-crushed results varies the organic carbon balance by 5.5%. Thus, it is important to obtain a representative sample of the spent shale and then crush and riffle the sample without changing its composition.

To insure good sampling and analyses of the spent shale, the following procedural and equipment changes have been instituted:

1. Sample size has been increased from 1% to 3%.

2. The spent shale sample crushing has been modified to prevent selective loss of dust.

d. Gas Product

(1) Leak at Top of Retort

Before Run 529 a hot-wire anemometer was used to check the flow of gas in the top of the raw shale standpipe and significant flow of air was detected as summarized below:

| Mean Retort Top Pressure Inches $H_2O$ | ≈Measured Velocity FPM | Calculated Air Flow SCFH |
|---|---|---|
| -.1 | 15 | 221 |
| -.2 | 25 | 368 |
| -.3 | 30 | 485 |

Since the policy had been to maintain a slight vacuum at the top of the retort, there had been an unaccounted

Table 6

Comparison of Spent Shale Before and After Retort Sample Crusher

| Run No. | Lab. No. | FA | $CO_2$ | Ash | $C_{tot}$ | H | $C_{org}$ |
|---|---|---|---|---|---|---|---|
| 541 B* | 1368 | -- | 17.3 | 80.6 | 7.33 | 0.18 | 2.61 |
| A | 1369 | 0.5 | 15.8 | 82.1 | 6.81 | 0.19 | 2.50 |
| 544 B | 1385 | 0.0 | 11.0 | 88.2 | 4.72 | 0.10 | 1.72 |
| A | 1388 | -- | 11.3 | 88.0 | 4.84 | 0.11 | 1.76 |
| 545 B | 1391 | 0.0 | 12.4 | 86.3 | 5.50 | 0.11 | 2.11 |
| A | 1406 | -- | 11.7 | 87.2 | 5.29 | 0.12 | 2.07 |
| 546 B | 1408 | 0.0 | 10.7 | 88.0 | 4.96 | 0.08 | 2.04 |
| A | 1412 | -- | 10.6 | 87.9 | 4.92 | 0.10 | 2.03 |
| 547 B | 1414 | 0.0 | 8.63 | 90.7 | 4.43 | 0.14 | 2.07 |
| A | 1417 | -- | 8.94 | 90.2 | 4.62 | 0.10 | 2.18 |
| 549 B | 1420 | 0.0 | 11.8 | 86.9 | 4.95 | 0.12 | 1.73 |
| A | 1424 | -- | 11.8 | 86.9 | 4.98 | 0.11 | 1.76 |
| 550 B | 1426 | 0.0 | 9.89 | 89.0 | 4.84 | 0.12 | 2.14 |
| A | 1429 | -- | 11.6 | 87.0 | 5.25 | 0.12 | 2.08 |
| 551 B | 1432 | 0.0 | 12.0 | 86.7 | 5.44 | 0.15 | 2.16 |
| A | 1436 | -- | 12.8 | 85.9 | 5.52 | 0.12 | 2.13 |

*"B" is before the retort crushing (the laboratory crushed sample) and "A" is after the retort crusher

for air leak into the system which could easily amount to as much as 500 SCF/T. This size leak would result in calculating the vent gas rate about 650 SCF/T low and thus the carbon balance would be low by about 3%.

In order to prevent this leak, 10 CFM of vent gas, cooled to 60° to 70° F to remove moisture which would corrode the raw shale sleeve, was piped to the top of the raw shale standpipe. The retort top pressure was maintained slightly positive so that less than 10 CFM of vent gas would leak through the star feeder the remainder would recirculate through the recovery system.

Additional star feeders will be purchased to permit the installation of pressure balance gas seals using vent gas at the top and bottom of Retort No. 1.

(2) Gas Sampling

(a) Larger Gas Sample for Condensate Determination

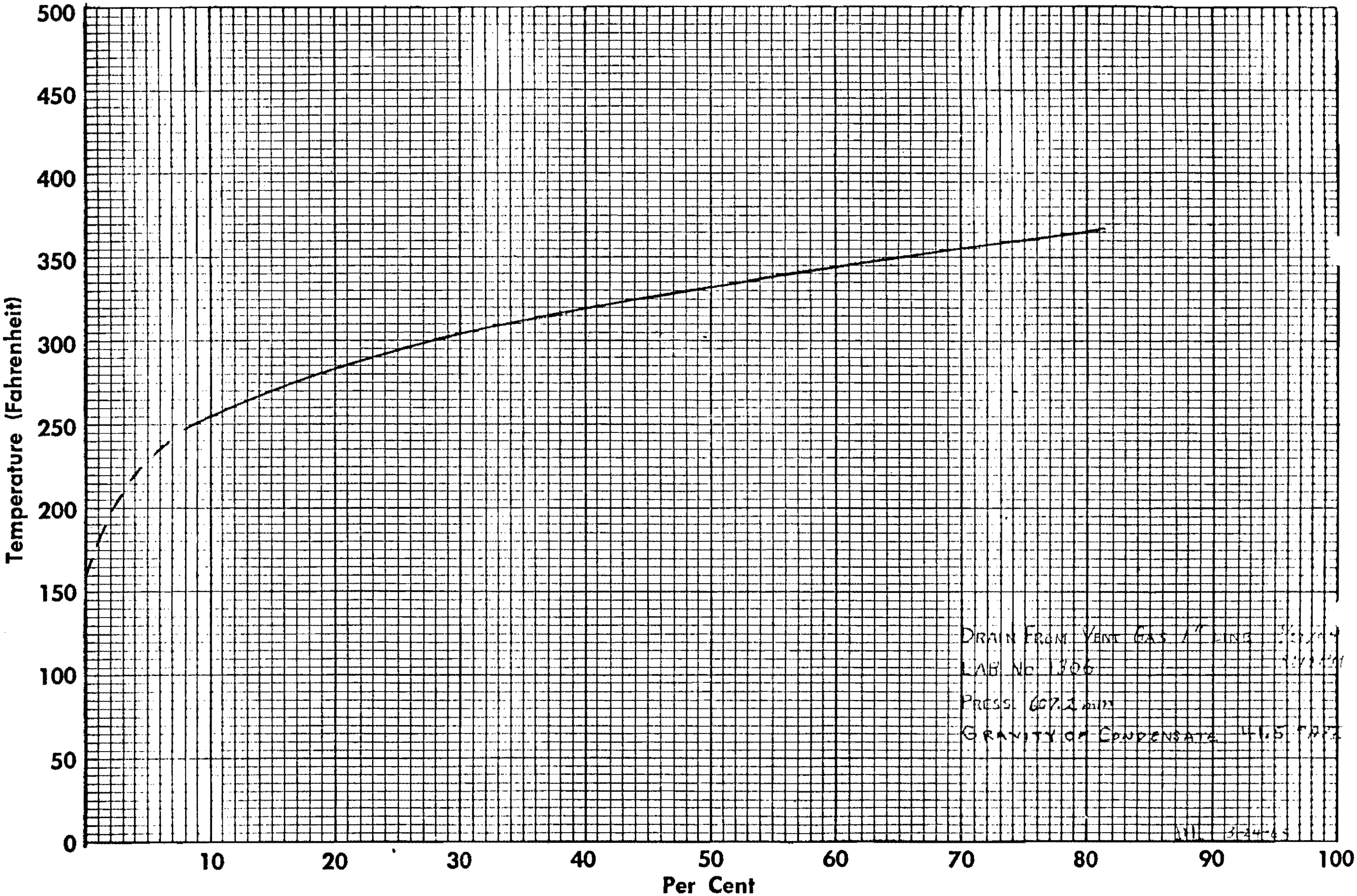
The 10 CFM vent gas purge is obtained from the total recycle line by means of an isokinetic gas sampler in order to assure the most representative gas sample possible. A glass section surrounding the sample tube permits observing whether oil is flowing along the wall and also if a mist is present. A double liquid trap is also provided at the outlet of the 60° F vent gas cooler to provide quantitative measurement of water and oil condensed. Nearly two quarts of oil condensate per run have been collected for the low yield runs from the 10 CFM purge gas. This condensate has increased organic carbon balance by as much as 2 wt %. On the low yield runs this condensate appears to be primarily a naphtha, as shown by the distillation curve and the 42° API gravity shown on Figure 2.

A glass pitot type sampler was installed on the dry vent gas purge line to obtain a composite sample during the runs. This change in sample location was made during Run 538. The 3/8-inch copper sample line between the retort and the gas holder in analytical, is heat traced, and is held at about 150° F to prevent any condensation. The heating value of the gas increased about 25 Btu/SCF after this change, indicating more hydrocarbons than were measured before the change.

Figure 2

Liquid Compensate From 60° Vent Gas Purge Condenser

## DISTILLATION CHART



The gas holder at analytical was inspected for signs of an oil film in the inside, which would indicate condensation of heavy hydrocarbons; and none was found.

(b) Dry Ice - Acetone Trap Analyses

A small sample of the vent gas after 70° F cooling was passed through a -110° F dry ice acetone trap and the condensate measured. A summary of the results follow:

RESULTS OF COLD-TRAPPING GAS SAMPLE AT THE LABORATORY

| Run No. | Pounds Material Collected/MCF Gas: Organic | Water |
|---|---|---|
| 526 | 1.05 | 1.3 |
| 528 | 1.02 | 0.7 |
| 529 | 0.98 | 3.6 |
| 530 | 0.74 | 0.3 |

The oil condensate gravity was about 73° API indicating primarily light naphtha. Assuming that the organic liquid is 81% C, the average C equivalent for the runs above is 0.75 lbs C/MCF gas. This, added to the 11.8 lbs C/MCF in the gas after the trap, would total about 12.6 lbs C/MCF gas, which is approximately the usual content, which indicates that these hydrocarbons are included in the normal carbon analysis of the gas.

(c) Comparison of Samples at the Retort Versus From the Line to the Gas Holder in the Analytical Building

As shown below, there is no significant difference in the major components by chromatograph analyses, whether sampling is directly at the retort or accomplished through a transfer line. However, the consistently higher "others" could account for more carbon in the gas. This data is further justification for relocating the sampling spot.

COMPARISONS OF GAS SAMPLES TAKEN AT THE RETORT AND THE LABORATORY
RUN 530

| | March 18 (4:45 p.m.) | | March 18 (8:30 p.m.) | |
|---|---|---|---|---|
| Component | Lab | Retort | Lab | Retort |
| $CO_2$ | 26.7 | 25.5 | 25.7 | 25.4 |
| $O_2$ | 0.6 | 0.3 | 0.7 | 0.7 |
| $N_2$ | 60.2 | 59.0 | 60.2 | 59.3 |
| $CH_4$ | 1.5 | 1.7 | 1.5 | 1.8 |
| CO | 5.3 | 5.7 | 5.4 | 5.6 |
| $H_2$ | 4.2 | 5.0 | 5.1 | 5.3 |
| Ar | 0.7 | 0.7 | 0.7 | 0.7 |
| Other | 0.8 | 1.7 | 0.7 | 1.2 |

(d) Grab Sample

A sample was taken from the vent gas purge line by means of a removable iron pipe with a gate valve at each end during Run 538.

This one preliminary experiment indicates that comparable results can be obtained on a gas obtained close to the retort or at the laboratory. Also that gas lines at the retort are probably coated with a thin layer of oil and water which can be driven off by heating. The results are summarized below:

| | Total # C/MSCF | Total # H/MSCF |
|---|---|---|
| Purged cold with oxygen for 1 hr. | 12.2 | 1.10 |
| Purged hot with oxygen for 1 1/2 hr. | 13.9 | 2.73 |
| Purged hot with oxygen for addn'l 2 hrs. | 14.1 | 3.47 |
| Run 538 normal gas sample | 12.5 | 0.87 |

(3) Analysis of Gas at Bottom of Retort

The material balance calculation determines the vent gas make by a nitrogen balance which assumes the leak out at the bottom star feeder is the same composition as the vent gas. There was some speculation that it may pick up hydrocarbons or $CO_2$ from the spent shale. Therefore, carbon analyses were made on spot samples taken from between the turntable and the bottom star feeder. As shown below the carbon is lower than that measured in the normal sample.

COMPARISONS OF C-H RESULTS ON GAS SAMPLES TAKEN AT THE RETORT AND THE LABORATORY

| | | C, lbs/MSCF | | H, lbs/MSCF | |
|---|---|---|---|---|---|
| Run No. | Date & Time | Btu of Retort | Normal | Btu of Retort | Normal |
| 531 | 3/20 11:30 p.m. | 11.9 | 13.5 | 0.84 | 0.92 |
| 532 | 3/21 11:50 p.m. | 11.8 | 12.7 | 0.81 | 0.93 |

Since these two comparisons are only based on spot samples definite conclusions can not be reached. If the mean organic carbon balances and the gas appear to be too high on future runs, this potential spot for carbon adsorption by the spent shale will be reappraised. The 1 lb. C/MSCF difference between the carbon content of the vent gas and the leak at the star feeder could result in overstating the organic carbon balance by about 2% when there is a large leak at the star feeder.

(4) Reappraisal of Carbon-Hydrogen Test for Gas Sample

(a) Effect of Water Adsorber

To show whether the magnesium perchlorate dryer which precedes the combustion train on the C-H apparatus, is extracting some organic material, samples were run with and without the magnesium perchlorate dryer. There is no evidence to show that the dryer extracts organic material from the gas; on the contrary, as shown below, there seems to be a slight negative bias when the dryer is not used. Further work will be done to explain this anomaly.

| | lb/MSCF Gas | | | |
|---|---|---|---|---|
| | With Dryer | | Without Dryer | |
| Run No. | C | H | C | H |
| 499 | 12.6 | 0.75 | 12.4 | 0.96 |
| 526 | 12.4 | 0.96 | 12.2 | 1.08 |
| 527 | 12.1 | 0.90 | 11.7 | 0.94 |
| 528 | 12.0 | 0.77 | 12.2 | 0.83 |
| 530 | 11.9 | 0.74 | 11.6 | 0.73 |

(b) Effect of Oxygen Purging and Heating of Gas Sample Container

i. It was efficacious to purge a sample from a sampling tube into the C-H combustion tube with $O_2$.

ii. Heating the sample tube would release any hydrocarbons that may have been adsorbed on the glass.

Therefore the following experiment was made to measure the effects of $O_2$ purge and heating:

A sample was transferred from a gas holder to a clear dry evacuated sample tube.

This sample was purged from the cold tube with a stream of oxygen at 80 ml/min. After two intervals, the tube was heated to about 150° F and the $O_2$ purge continued for two more intervals. The tube was cooled, and additional purging was completed.

The following table shows that oxygen purging, although a more lengthy process, gives essentially the same results as brine displacement, and that heating the tube gives no additional, possibly adsorbed, carbon.

RESULTS OF OXYGEN PURGING AND HEATING OF RECYCLE GAS SAMPLE CONTAINER (GAS SAMPLE FROM RUN 544)

| Container Conditions | Purge Time, min. | Totals | | | |
|---|---|---|---|---|---|
| | | mg $CO_2$ | # C/MSCF | mg $H_2O$ | # H/MSCF |
| Normal Brine Purged Sample | | -- | 12.9 | -- | 0.80 |
| Cold | 80 | 421.0 | 12.9 | 62.1 | 0.78 |
| Cold | 70 | -0.1 | 12.9 | 2.4 | 0.81 |
| 150° F | 60 | -0.9 | 12.9 | -0.7 | 0.82 |
| 150° F | 60 | -1.1 | 12.8 | 0.7 | 0.81 |
| Cold | 75 | 0.4 | 12.9 | 1.1 | 0.82 |
| Cold | 60 | -0.4 | 12.8 | -1.2 | 0.81 |

In order to be sure hydrocarbons were not adsorbed during the brine displacement of the gas from the sample bomb into the combustion tube several tests were made using mercury. The results summarized below show no significant evidence that the various displacement methods give different results.

EFFECT OF DIFFERENT TYPES OF DISPLACEMENT FOR THE DETERMINATION OF C-H IN RECYCLE GAS

| Sample | Hg Displaced | | Brine Displaced | |
|---|---|---|---|---|
| | lbs C/MSCF | lbs H/MSCF | lbs C/MSCF | lbs H/MSCF |
| "B Standard" | 10.1 | 0.23 | 9.9 | 0.29 |
| Run 544 | 13.0 | 0.73 | 13.0 | 0.80 |
| Run 549 | 13.2 | 0.52 | 13.2 | 0.69 |

(c) <u>Effect of Combustion Oxygen Rate on C-H Analysis</u>

The effect of oxygen rate in the carbon determination was evaluated and no evidence of any significant difference was found as shown below; however, more data will be collected to further substantiate this.

| Sample | O2 Rate 25 ml/min C | H | O2 Rate 50 ml/min C | H |
|---|---|---|---|---|
| Lab. No. 1374 | 12.4 | 1.19 | 12.6 | 1.22 |
| "B Standard" | 9.9 | 0.29 | 9.9 | 0.30 |

All the tests on the equipment and procedures for measuring the carbon - hydrogen contents of the retort vent gas indicate they are reliable and adequately precise when a representative sample is obtained.

(5) Calibration of Air Orifice Meter

The air rate is the basis for calculating the vent gas and, therefore, a very important term in the carbon balance, as well as an important process variable. It was calibrated prior to Run 530.

The calibration technique used was that of passing a known quantity of air, determined by weighing bottled air cylinders before and after a calibration run, through the orifice meter and recording the pressure drop across the meter. Static pressure and temperature measurements were also made.

Results of the first calibration showed that the actual air rate was 11% higher than that indicated by the orifice meter. At the end of Run 537, the meter run was disassembled, inspected, rebuilt, and recalibrated. Results of the second calibration indicate that the actual air rate for the reinstalled meter was 6% higher than the rate indicated by the meter.

An American Meter Co. Model 60B bellows type dry gas meter has been purchased and installed in the air line in series with the orifice meter.

Calibrations of the air orifice against the dry gas meter check the 6% factor obtained by the air cylinder method. This correction has been applied to runs after Run 537.

(6) Use of Leak Detector For Checking For Gas Leaks Out of the Retort

Leaks of hydrocarbon vapor from the retort directly reduce carbon balances. The No. 1 Retort was checked for hydrocarbon leakage on March 16, 1965, using the MSA Explosimeter Gas Indicator Model No. 2A. This device indicates the presence of combustible material by catalytically oxidizing a sample and measuring temperature increase with a thermocouple. Checks for hydrocarbon leakage were made at several locations.

No outward leakage was detected at the top of the raw shale feed standpipe. No hydrocarbon was found mid-way down on the raw shale feed standpipe.

A small amount of hydrocarbon was detected in the vapor space of product storage tank and decanter drum, as expected.

A high concentration of combustibles was present under the conveyor cover at the bottom of the retort where the spent shale is removed. This was expected also, since recycle gas leaks out of the star feeder at that point.

Leakage was checked at each thermowell nipple and fitting on the retort shell, but no hydrocarbon leakage was detected.

In summary, no leakage of hydrocarbon was found from the No. 1 Retort vessel except the known leak at the star feeder. The small amount of hydrocarbon detected in oil product test tank vapor space was insufficient to cause carbon balance problems. This conclusion was confirmed by later laboratory weathering tests. Later testing indicated air was leaking in, rather than hydrocarbon leaking out.

SUMMARY SHEET

| RUN NUMBER(1) | 525 | 526 | 527 | 528 | 529 |
|---|---|---|---|---|---|
| DATE STARTED | 3/15/65 | 3/16/65 | 3/16/65 | 3/17/65 | 3/18/65 |
| LENGTH OF RUN, hours | 6 | 8 | 8 | 8 | 8 |
| RETORT TYPE NUMBER | R-H | R-H | R-H | R-H | R-H |
| OIL RECOVERY SYSTEM NUMBER | M-10 | M-10 | M-10 | M-10 | M-10 |
| OPERATING CONDITIONS: | | | | | |
| Raw Shale, lbs/(hr)($ft^2$) | 593 | 712 | 701 | 488 | 640 |
| Fischer Assay, Gal/Ton RS | 27.5 | 29.4 | 29.3 | 31.3 | 30.4 |
| Nom. Size Range, inches | 3/4-1½ | 3/4-1½ | 3/4-1½ | 3/4-1½ | 3/4-1½ |
| Avg. Part. Diam., inches | 0.66 | 0.74 | 0.63 | 0.76 | 0.71 |
| Air, SCF/Ton RS | 5039 | 4985 | 5129 | 5595 | 5651 |
| Tot. Recycle, SCF/Ton RS(wet)(2) | 12418 | 12464 | 12580 | 12230 | |
| Dilution Gas, SCF/Ton RS (wet) | 0 | 0 | 0 | 0 | 0 |
| Propane, SCF/Ton RS | 0 | 0 | 0 | 0 | 0 |
| Brine, Gal/Ton RS | 0 | 0 | 0 | 0 | 0 |
| Air Temp. Entering Retort, °F | 66 | 77 | 66 | 67 | 57 |
| Bed Hgt. Above Air Dist., ft | 9 | 7 | 7 | 7 | 7 |
| OPERATING DATA: | | | | | |
| Offgas Temperature, °F | 115 | 172 | 171 | 144 | 180 |
| Recycle Gas Temperature, °F | 157 | 163 | 163 | 155 | 156 |
| Spent Shale Temperature, °F | 237 | 243 | 243 | 248 | 201 |
| Avg. Retort $\Delta P$, in. $H_2O$/ft | 1.31 | 1.94 | 1.84 | 1.44 | 1.94 |
| $\Delta P$ Above Air Dist. in $H_2O$/ft | 1.24 | 2.37 | 2.11 | 1.24 | 2.37 |
| Overall Oper. Performance | GOOD | GOOD | GOOD | GOOD. | GOOD |
| DUST lb/hr | 0 | 0 | 0 | 0 | 2 |
| PRODUCTS RECOVERED: | | | | | |
| Oil Collected, vol % RSFA | 82.9 | 76.4 | 79.8 | 85.1 | 84.4 |
| Oil Lost as Mist, vol % RSFA | 0.0 | 0.1 | 0.0 | 0.1 | 0.0 |
| Oil in Spent Shale, vol % RSFA | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Total Oil Meas., vol % RSFA | 82.9 | 76.5 | 79.8 | 85.2 | 84.4 |
| Total Water, lbs/Ton RS | 65.5 | 60.3 | 60.2 | 59.4 | 65.3 |
| Calc. Dry Vent Gas, SCF/Ton RS | 6509 | 6327 | 6551 | 7287 | 7242 |
| Mineral $CO_2$ Decomposed, % | 41.6 | 32.8 | 33.2 | 45.5 | 42.9 |
| MATERIAL BALANCES: | | | | | |
| Ash, wt % (measured) | 99.4 | 99.3 | 98.6 | 99.3 | 96.1 |
| Basis for Yields & Mat'l. Bal. | RS | RS | SS | RS | SS |
| Overall Balance, wt % | 98.2 | 98.1 | 98.8 | 97.6 | 98.9 |
| Organic Carbon Balance, wt % | 92.5 | 92.0 | 93.7 | 89.0 | 93.3 |
| Total Carbon Balance, wt % | 94.6 | 94.1 | 95.4 | 91.7 | 95.1 |
| Organic Hydrogen Balance, wt % | 99.9 | 96.7 | 94.8 | 93.0 | 94.7 |
| Water Balance, wt % | 90.0 | 89.0 | 101.1 | 85.6 | 92.1 |
| Gas Loss, SCF/Ton RS ~~(dry)~~ WET | 3136 | 3145 | 3301 | 2314 | 4090 |
| HEAT BALANCE: | | | | | |
| Heat of Combustion, MBtu/Ton RS | 478.6 | 467.7 | 462.9 | 513.2 | 484.4 |
| Unaccounted Heat, MBtu/Ton RS | 110.4 | 59.6 | 51.3 | 42.1 | 2.2 |
| SHALE OIL PROPERTIES: | | | | | |
| Gravity, °API | 20.2 | 18.9 | 19.5 | 19.4 | 19.7 |
| Ramsbottom Carbon, wt % | 2.13 | 2.13 | 2.25 | 2.32 | 2.21 |
| Ash, wt % | 0.02 | 0.02 | 0.02 | 0.04 | 0.10 |
| GAS PROPERTIES (DRY): | | | | | |
| Moisture, lbs/MSCF of dry gas | 4.7 | 6.5 | 6.4 | 5.6 | 8.4 |
| Gross Heating Value, Btu/SCF | 98.2 | 127.2 | 115 | 96 | 96.9 |
| $O_2$ vol % | 0.7 | 0.8 | 1.0 | 0.9 | 1.9 |
| $CO_2$ vol % | 25.4 | 22.5 | 23.2 | 25.7 | 24.5 |
| SPENT SHALE: | | | | | |
| Fischer Assay, Gal/Ton SS | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Organic Carbon, wt % | 1.42 | 1.81 | 2.23 | 1.86 | 2.14 |

(1) Retort No. 1 runs - no prefix; Retort No. 2 runs - "B" prefix.
(2) Measure Recycle + Dilution Gas Rate Minus Wet Gas Loss

SUMMARY SHEET

| | | | | | |
|---|---|---|---|---|---|
| RUN NUMBER(1) | 530 | 531 | 532 | 533 | 534 |
| DATE STARTED | 3/18/65 | 3/20/65 | 3/21/65 | 3/21/65 | 3/22/65 |
| LENGTH OF RUN, hours | 8 | 8 | 8 | 8 | 8 |
| RETORT TYPE NUMBER | R-H | R-I | R-I | R-I | R-I |
| OIL RECOVERY SYSTEM NUMBER | M-10 | M-10 | M-10 | M-10 | M-10 |
| OPERATING CONDITIONS: | | | | | |
| Raw Shale, lbs/(hr)($ft^2$) | 643 | 651 | 660 | 655 | 660 |
| Fischer Assay, Gal/Ton RS | 28.4 | 29.6 | 29.4 | 30.6 | 31.2 |
| Nom. Size Range, inches | 3/4-1½ | 3/4-1½ | 3/4-1½ | 3/4-1½ | 3/4-1½ |
| Avg. Part. Diam., inches | 0.68 | 0.60 | 0.65 | 0.68 | 0.66 |
| Air, SCF/Ton RS | 5571 | 5525 | 5583 | 5450 | 5412 |
| Tot. Recycle, SCF/Ton RS (wet)(2) | 12503 | 12427 | 12844 | 12389 | 12048 |
| Dilution Gas, SCF/Ton RS (wet) | 0 | 0 | 0 | 0 | 0 |
| Propane, SCF/Ton RS | 0 | 0 | 0 | 0 | 0 |
| Brine, Gal/Ton RS | 0 | 0 | 0 | 0 | 0 |
| Air Temp. Entering Retort, °F | 65 | 64 | 69 | 72 | 70 |
| Bed Hgt. Above Air Dist., ft | 7 | 7 | 7 | 7 | 7 |
| | | | | | |
| OPERATING DATA: | | | | | |
| Offgas Temperature, °F | 167 | 165 | 164 | 161 | 164 |
| Recycle Gas Temperature, °F | 157 | 157 | 159 | 160 | 158 |
| Spent Shale Temperature, °F | 208 | 204 | 214 | 216 | 207 |
| Avg. Retort $\Delta P$, in. $H_2O$/ft | 2.00 | 1.92 | 1.92 | 1.76 | 1.76 |
| $\Delta P$ Above Air Dist. in $H_2O$/ft | 2.40 | 2.30 | 2.11 | 2.01 | 2.11 |
| Overall Oper. Performance | GOOD | GOOD | GOOD | GOOD | GOOD |
| | | | | | |
| PRODUCTS RECOVERED: | | | | | |
| Oil Collected, vol % RSFA | 84.0 | 84.4 | 87.5 | 86.1 | 85.2 |
| Oil Lost as Mist, vol % RSFA | 0.0 | 0.0 | 0.0 | 1.5 | 1.2 |
| Oil in Spent Shale, vol % RSFA | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Total Oil Meas., vol % RSFA | 84.0 | 84.4 | 87.5 | 87.6 | 86.4 |
| Total Water, lbs/Ton RS | 69.7 | 65.7 | 64.3 | 61.4 | 64.9 |
| Calc. Dry Vent Gas, SCF/Ton RS | 7416 | 7507 | 7177 | 7278 | 7327 |
| Mineral $CO_2$ Decomposed, % | 44.2 | 40.5 | 37.9 | 32.3 | 38.0 |
| MATERIAL BALANCES: | | | | | |
| Ash, wt % (measured) | 95.7 | 96.5 | 97.9 | 97.3 | 98.7 |
| Basis for Yields & Mat'l. Bal. | SS | SS | SS | SS | RS |
| Overall Balance, wt % | 100.5 | 100.3 | 99.7 | 100.4 | 99.6 |
| Organic Carbon Balance, wt % | 96.6 | 98.7 | 99.2 | 99.9 | 102.0 |
| Total Carbon Balance, wt % | 97.5 | 99.0 | 99.4 | 99.9 | 101.5 |
| Organic Hydrogen Balance, wt % | 99.3 | 95.3 | 96.9 | 91.9 | 100.7 |
| Water Balance, wt % | 116.2 | 112.4 | 105.8 | 152.9 | 109.5 |
| Gas Loss, SCF/Ton RS ~~(dry)~~ WET | 4450 | 4557 | 3936 | 4490 | 4743 |
| HEAT BALANCE: | | | | | |
| Heat of Combustion, MBtu/Ton RS | 512.7 | 497.4 | 495.4 | 473.0 | 493.5 |
| Unaccounted Heat, MBtu/Ton RS | 52.1 | 49.2 | 59.3 | 76.5 | 54.2 |
| SHALE OIL PROPERTIES: | | | | | |
| Gravity, °API | 19.8 | 19.4 | 19.4 | 19.4 | 19.4 |
| Ramsbottom Carbon, wt % | 2.09 | 2.37 | 2.37 | 2.39 | 2.41 |
| Ash, wt % | 0.04 | 0.05 | 0.04 | 0.03 | 0.03 |
| GAS PROPERTIES (DRY): | | | | | |
| Moisture, lbs/MSCF of dry gas | 6.1 | 6.8 | 7.1 | 7.0 | 7.2 |
| Gross Heating Value, Btu/SCF | 106.1 | 119.2 | 120.6 | 131.3 | 126.9 |
| $O_2$ vol % | 0.4 | 0.5 | 0.4 | 0.5 | 0.3 |
| $CO_2$ vol % | 26.5 | 25.0 | 24.8 | 24.3 | 24.4 |
| SPENT SHALE: | | | | | |
| Fischer Assay, Gal/Ton SS | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Organic Carbon, wt % | 1.54 | 1.78 | 1.55 | 1.66 | 1.86 |

(1) Retort No. 1 runs - no prefix; Retort No. 2 runs - "B" prefix.

(2) Measure Recycle + Dilution Gas Rate Minus Wet Gas Loss

JWH 352

SUMMARY SHEET

| | | | |
|---|---|---|---|
| RUN NUMBER(1) | 535 | 536 | 537 |
| DATE STARTED | 3/22/65 | 3/23/65 | 3/24/65 |
| LENGTH OF RUN, HOURS | 8 | 8 | 8 |
| RETORT TYPE NUMBER | R-I | R-I | R-I |
| OIL RECOVERY SYSTEM NUMBER | M-10 | M-10 | M-10 |
| OPERATING CONDITIONS: | | | |
| Raw Shale, lbs/(hr)(ft$^2$) | 662 | 666 | 678 |
| Fischer Assay, Gal/Ton RS | 27.9 | 27.6 | 31.2 |
| Nom. Size Range, inches | 3/4-1 1/2 | 3/4-1 1/5 | 3/4-1 1/2 |
| Avg. Part. Diam., inches | 0.69 | 0.633 | 0.686 |
| Air, SCF/Ton RS | 5455 | 5416 | 5327 |
| Total Recycle, SCF/Ton RS(2) (wet) | 11733 | 11556 | 11746 |
| Dilution Gas, SCF/Ton RS (wet) | 0 | 0 | 0 |
| Propane, SCF/Ton RS | 0 | 0 | 0 |
| Brine, Gal/Ton RS | 0 | 0 | 0 |
| Air Temp. Entering Retort, °F | 62 | 60 | 58 |
| Bed Hgt. Above Air Dist., ft | 6 | 6 | 6 |
| | | | |
| OPERATING DATA: | | | |
| Offgas Temperature, °F | 197 | 211 | 196 |
| Recycle Gas Temperature, °F | 163 | 164 | 162 |
| Spent Shale Temperature, °F | 205 | 207 | 194 |
| Avg. Retort ΔP, in. $H_2O$/Ft | 1.74 | 1.57 | 1.53 |
| ΔP Above Air Dist. in $H_2O$/Ft | 2.06 | 1.71 | 1.68 |
| Overall Oper. Performance | GOOD | GOOD | GOOD |
| | | | |
| PRODUCTS RECOVERED: | | | |
| Oil Collected, vol % RSFA | 76.7 | 78.2 | 82.5 |
| Oil Lost as Mist, vol % RSFA | 0.1 | 0.1 | 1.8 |
| Oil in Spent Shale, vol % RSFA | 0.0 | 0.0 | 0.0 |
| Total Oil Meas., vol % RSFA | 76.8 | 78.3 | 84.3 |
| Total Water, lbs/Ton RS | 95.9 | 100.6 | 80.9 |
| Calc. Dry Vent Gas, SCF/Ton RS | 7164 | 7009 | 6997 |
| Mineral $CO_2$ Decomposed, % | 39.4 | 33.8 | 33.1 |
| | | | |
| MATERIAL BALANCES: | | | |
| Ash, wt % (measured) | 99.7 | 100.2 | 99.2 |
| Basis for Yields & MB | RS | RS | RS |
| Overall, wt % | 99.6 | 99.9 | 100.2 |
| Organic Carbon, wt % | 91.4 | 93.2 | 97.8 |
| Total Carbon, wt % | 93.8 | 95.0 | 98.4 |
| Organic Hydrogen, wt % | 88.1 | 97.9 | 92.6 |
| Water, wt % | 153.4 | 172.8 | 166.8 |
| Gas Loss, SCF/Ton RS ~~(dry)~~ WET | 5214 | 5402 | 4777 |
| HEAT BALANCE: | | | |
| Heat of Comb., MBtu/Ton RS | 501.5 | 495.0 | 469.2 |
| Unaccounted, MBtu/Ton RS | 50.4 | 65.9 | 51.2 |
| SHALE OIL PROPERTIES: | | | |
| Gravity, °API | 19.2 | 19.0 | 19.2 |
| Ramsbottom Carbon, wt % | 2.51 | 2.61 | 2.69 |
| Ash, wt % | 0.01 | 0.01 | 0.09 |
| GAS PROPERTIES (DRY) | | | |
| Moisture lbs/MSCF | 12.5 | 13.8 | 10.3 |
| Gross Heating Value Btu/SCF | 98.3 | 109.6 | 127.9 |
| $O_2$ vol % | 0.5 | 0.4 | 0.5 |
| $CO_2$ vol % | 25.0 | 24.0 | 24.6 |
| SPENT SHALE: | | | |
| Fischer Assay, Gal/Ton SS | 0.0 | 0.0 | 0.0 |
| Organic Carbon, wt % | 1.51 | 1.65 | 1.71 |

(1) Retort No. 1 runs - no suffix; Retort No. 2 runs - "B" suffix.
(2) Meas. Recycle + Dilution Gas Rate Minus Wet Gas Loss

## SUMMARY SHEET

| | | | | | |
|---|---|---|---|---|---|
| RUN NUMBER(1) | 538 | 539 | 540 | 541 | 542 |
| DATE STARTED | 3-26 | 3-27 | 3-27 | 3-28 | 3-28 |
| LENGTH OF RUN, hours | 8 | 12 | 12 | 12 | 12 |
| RETORT TYPE NUMBER | R-I | R-I | R-I | R-I | R-I |
| OIL RECOVERY SYSTEM NUMBER | M-10 | M-10 | M-10 | M-10 | M-10 |
| **OPERATING CONDITIONS:** | | | | | |
| Raw Shale, lbs/(hr)(ft$^2$) | 644 | 710 | 726 | 720 | 748 |
| Fischer Assay, Gal/Ton RS | 27.4 | 27.6 | 29.7 | 30.6 | [illegible] |
| Nom. Size Range, inches | 3/4 - 1½ | 3/4 - 1½ | 3/4 - 1½ | 3/4 - 1½ | 3/4 - 1½ |
| Avg. Part. Diam., inches | 0.518 | 0.690 | 0.710 | 0.718 | 0.700 |
| Air, SCF/Ton RS | 5806 | 5248 | 5250 | 5320 | 5130 |
| Tot. Recycle, SCF/Ton RS(wet) (2) | 10737 | 9703 | 9487 | 10691 | 10571 |
| Dilution Gas, SCF/Ton RS (wet) | — | — | — | — | — |
| Propane, SCF/Ton RS | — | — | — | — | — |
| Brine, Gal/Ton RS | — | — | — | — | — |
| Air Temp. Entering Retort, °F | 63 | 63 | 66 | 66 | 61 |
| Bed Hgt. Above Air Dist., ft | 6.0 | 6.0 | 6.0 | 6.0 | 6.0 |
| | | | | | |
| **OPERATING DATA:** | | | | | |
| Offgas Temperature, °F | 169 | 180 | 190 | 171 | 165 |
| Recycle Gas Temperature, °F | 164 | 163 | 170 | 169 | 166 |
| Spent Shale Temperature, °F | 199 | 218 | 285 | 270 | 280 |
| Avg. Retort ΔP, in. $H_2O$/ft | 1.64 | 1.55 | 1.44 | 1.43 | 1.50 |
| ΔP Above Air Dist. in $H_2O$/ft | 1.40 | 1.38 | 1.25 | 1.19 | 1.27 |
| Overall Oper. Performance | GOOD | GOOD | GOOD | GOOD | GOOD |
| | | | | | |
| **PRODUCTS RECOVERED:** | | | | | |
| Oil Collected, vol % RSFA | 82.7 | 79.0 | 77.6 | 78.0 | 80.8 |
| Oil Lost as Mist, vol % RSFA | 2.8 | 2.5 | 2.4 | 1.9 | 1.4 |
| Oil in Spent Shale, vol % RSFA | 0.0 | 0.0 | 0.0 | 1.4 | 0.0 |
| Total Oil Meas., vol % RSFA | 85.5 | 81.5 | 79.0 | 81.5 | 82.2 |
| Total Water, lbs/Ton RS | 76.0 | 68.3 | 84.1 | 61.2 | 47.5 |
| Calc. Dry Vent Gas, SCF/Ton RS | 7765 | 6894 | 6607 | 6873 | 6606 |
| Mineral $CO_2$ Decomposed, % | 43.4 | 28.1 | 20.3 | 20.9 | 23.8 |
| **MATERIAL BALANCES:** | | | | | |
| Ash, wt % (measured) | 97.9 | 97.5 | 99.1 | 99.2 | 98.3 |
| Basis for Yields & Mat'l. Bal. | SS | SS | SS | SS | SS |
| Overall Balance, wt % | 100.6 | 101.0 | 101.2 | 99.9 | 99.2 |
| Organic Carbon Balance, wt % | 101.7 | 105.1 | 100.5 | 99.8 | 100.0 |
| Total Carbon Balance, wt % | 101.2 | 103.6 | 100.4 | 99.8 | 100.0 |
| Organic Hydrogen Balance, wt % | 99.2 | 104.3 | 91.1 | 92.7 | 99.4 |
| Water Balance, wt % | 148.0 | 114.1 | 159.8 | 141.8 | 82.0 |
| Gas Loss, SCF/Ton RS (dry) | 6383 | 5944 | 6065 | 4807 | 4489 |
| **HEAT BALANCE:** | | | | | |
| Heat of Combustion, MBtu/Ton RS | 557.9 | 475.0 | 466.0 | 467.1 | 470.2 |
| Unaccounted Heat, MBtu/Ton RS | 108.7 | 86.5 | 111.8 | 101.6 | 82.9 |
| **SHALE OIL PROPERTIES:** | | | | | |
| Gravity, °API | 18.8 | 19.0 | 19.0 | 19.0 | 19.2 |
| Ramsbottom Carbon, wt % | - | 2.56 | 2.59 | 2.74 | 2.99 |
| Ash, wt % | — | 0.02 | 0.04 | 0.02 | 0.02 |
| **GAS PROPERTIES (DRY):** | | | | | |
| Moisture, lbs/MSCF of dry gas | 9.7 | 9.1 | 12.0 | 7.83 | 6.7 |
| Gross Heating Value, Btu/SCF | 125 | 150 | 146 | 159 | 155 |
| $O_2$ vol % | 0.4 | 1.0 | 0.9 | 0.6 | 0.6 |
| $CO_2$ vol % | 25.2 | 21.8 | 20.5 | 21.1 | 21.1 |
| **SPENT SHALE:** | | | | | |
| Fischer Assay, Gal/Ton SS | 0.0 | 0.0 | 0.0 | 0.5 | 0.0 |
| Organic Carbon, wt % | 1.99 | 2.36 | 2.82 | 2.50 | 2.24 |

(1) Retort No. 1 runs - no prefix; Retort No. 2 runs - "B" prefix.
(2) Measure Recycle + Dilution Gas Rate Minus Wet Gas Loss

OSRC-11

REVISED SPENT SHALE ANALYSIS & L.P. CALC.

4-13-65 [illegible]

## SUMMARY SHEET

| | | | | | |
|---|---|---|---|---|---|
| RUN NUMBER(1) | [illegible] | [illegible] | 545 | 546 | 547 |
| DATE STARTED | 3-30-65 | 3-30-65 | 3-31-65 | 4-1-65 | 4-2-65 |
| LENGTH OF RUN, hours | 12 | 12 | 12 | 11½ | 12 |
| RETORT TYPE NUMBER | R-I | R-I | R-I | R-I | R-I |
| OIL RECOVERY SYSTEM NUMBER | M-10 | M-10 | M-10 | M-10 | M-10 |
| OPERATING CONDITIONS: | | | | | |
| Raw Shale, lbs/(hr)(ft$^2$) | 592 | 597 | 571 | 568 | 572 |
| Fischer Assay, Gal/Ton RS | 29.4 | 29.0 | 30.2 | 29.7 | 31.0 |
| Nom. Size Range, inches | ¾-1½ | ¾-1½ | ¾-1½ | ¾-1½ | ¾-1½ |
| Avg. Part. Diam., inches | 0.67 | 0.676 | 0.707 | [illegible] | 0.686 |
| Air, SCF/Ton RS | 7100 | 6255 | 6479 | 6545 | 6600 |
| Tot. Recycle, SCF/Ton RS(wet)(2) | [illegible] | 9265 | 9446 | 9062 | 10451 |
| Dilution Gas, SCF/Ton RS (wet) | — | — | — | — | — |
| Propane, SCF/Ton RS | — | — | — | — | — |
| Brine, Gal/Ton RS | — | — | — | — | — |
| Air Temp. Entering Retort, °F | 74 | 67 | 75 | 71 | 62 |
| Bed Hgt. Above Air Dist., ft | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 |
| | | | | | |
| OPERATING DATA: | | | | | |
| Offgas Temperature, °F | 120 | 119 | 122 | 125 | 129 |
| Recycle Gas Temperature, °F | 152 | 158 | 161 | 160 | 150 |
| Spent Shale Temperature, °F | 320 | 280 | 230 | 226 | 209 |
| Avg. Retort ΔP, in. $H_2O$/ft | 1.43 | 1.15 | 1.09 | 1.19 | 1.64 |
| ΔP Above Air Dist. in $H_2O$/ft | — | — | 0.81 | — | 1.93 |
| Overall Oper. Performance | GOOD | GOOD | GOOD | GOOD | GOOD |
| | | | | | |
| PRODUCTS RECOVERED: | | | | | |
| Oil Collected, vol % RSFA | 88.0 | 89.8 | 91.3 | 88.4 | 90.1 |
| Oil Lost as Mist, vol % RSFA | 0.4 | 0.6 | 0.7 | 0.8 | .7 |
| Oil in Spent Shale, vol % RSFA | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Total Oil Meas., vol % RSFA | 88.4 | 90.4 | 92.0 | 89.2 | 90.8 |
| Total Water, lbs/Ton RS | 70.0 | 61.6 | 74.1 | 75.5 | 99.2 |
| Calc. Dry Vent Gas, SCF/Ton RS | [illegible] | 8410 | [illegible] | 8855 | 8986 |
| Mineral $CO_2$ Decomposed, % | [illegible] | 48.1 | 43.9 | 51.0 | 60.6 |
| MATERIAL BALANCES: | | | | | |
| Ash, wt % (measured) | 98.1 | 99.5 | 98.2 | 97.9 | 99.2 |
| Basis for Yields & Mat'l. Bal. | SS | SS | SS | SS | SS |
| Overall Balance, wt % | [illegible] | 99.8 | 101.2 | 101.1 | 100.0 |
| Organic Carbon Balance, wt % | [illegible] | [illegible] | 108.9 | 104.7 | 103.9 |
| Total Carbon Balance, wt % | [illegible] | 102.3 | 106.7 | 103.5 | 102.9 |
| Organic Hydrogen Balance, wt % | 101.0 | 97.7 | 96.8 | 101.1 | 103.7 |
| Water Balance, wt % | 112.4 | 115.4 | 235.6 | 150.7 | 123.4 |
| Gas Loss, SCF/Ton RS (dry) | 6475 | 6182 | 6412 | 7012 | 5622 |
| HEAT BALANCE: | | | | | |
| Heat of Combustion, MBtu/Ton RS | 656.4 | 556.8 | 544.6 | 580.8 | 598.2 |
| Unaccounted Heat, MBtu/Ton RS | 92.5 | 68.1 | 113.1 | 96.8 | 68.4 |
| SHALE OIL PROPERTIES: | | | | | |
| Gravity, °API | 19.2 | 19.5 | 19.6 | 19.7 | 20.5 |
| Ramsbottom Carbon, wt % | 2.18 | 2.31 | 2.50 | 2.40 | 1.93 |
| Ash, wt % | 0.02 | 0.02 | 0.01 | 0.01 | 0.01 |
| GAS PROPERTIES (DRY): | | | | | |
| Moisture, lbs/MSCF of dry gas | 6.1 | 7.5 | 7.2 | 7.2 | 4.0 |
| Gross Heating Value, Btu/SCF | 99.6 | 107.1 | 125.4 | 125.4 | 105.9 |
| $O_2$ vol % | 0.2 | 0.4 | 0.3 | 0.2 | 0.3 |
| $CO_2$ vol % | 28.0 | 26.9 | 26.5 | 22.1 | 26.8 |
| SPENT SHALE: | | | | | |
| Fischer Assay, Gal/Ton SS | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Organic Carbon, wt % | 1.21 | 1.74 | 2.12 | 2.04 | 1.92 |

(1) Retort No. 1 runs - no prefix; Retort No. 2 runs - "P" prefix.
(2) Measure Recycle + Dilution Gas Rate Minus Wet Gas Loss

OSRC-11

REVISED SPENT SHALE ANALYSIS & L.P. CALC.
4-13-65 RI

SUMMARY SHEET

| | | | | | |
|---|---|---|---|---|---|
| RUN NUMBER(1) | [illegible] | [illegible] | [illegible] | [illegible] | |
| DATE STARTED | | 4-4 | 4-5 | 4-5 | |
| LENGTH OF RUN, hours | | 12 | 8 | 8 | |
| RETORT TYPE NUMBER | | 2-I | 2-I | 2-I | |
| OIL RECOVERY SYSTEM NUMBER | | M-11 | M-11 | M-11 | |
| OPERATING CONDITIONS: | | | | | |
| Raw Shale, lbs/(hr)(ft$^2$) | | 599 | 603 | 597 | |
| Fischer Assay, Gal/Ton RS | | [illegible] | [illegible] | [illegible] | |
| Nom. Size Range, inches | | 3/4-1½ | 3/4-1½ | 3/4-1½ | |
| Avg. Part. Diam., inches | | 0.639 | 0.696 | 0.753 | |
| Air, SCF/Ton RS | | 5486 | 5434 | 5490 | |
| Tot. Recycle, SCF/Ton RS(wet)(2) | | 11656 | 11922 | 11717 | |
| Dilution Gas, SCF/Ton RS (wet) | | — | — | — | |
| Propane, SCF/Ton RS | | — | — | — | |
| Brine, Gal/Ton RS | | — | — | — | |
| Air Temp. Entering Retort, °F | | 50 | 60 | 50 | |
| Bed Hgt. Above Air Dist., ft | | 9.0 | [illegible] | [illegible] | |
| | | | | | |
| OPERATING DATA: | | | | | |
| Offgas Temperature, °F | | 122 | 97 | 142 | |
| Recycle Gas Temperature, °F | | 148 | 169 | 150 | |
| Spent Shale Temperature, °F | | 214 | 174 | 169 | |
| Avg. Retort ΔP, in. $H_2O$/ft | | 1.74 | 1.71 | 1.65 | |
| ΔP Above Air Dist. in $H_2O$/ft | | 1.97 | 2.01 | 1.83 | |
| Overall Oper. Performance | | GOOD | GOOD | [illegible] | |
| | | | | | |
| PRODUCTS RECOVERED: | | | | | |
| Oil Collected, vol % RSFA | | 88.1 | 84.1 | 86.6 | |
| Oil Lost as Mist, vol % RSFA | | 0.6 | .7 | 0.9 | |
| Oil in Spent Shale, vol % RSFA | | 0.0 | 0.0 | 0.0 | |
| Total Oil Meas., vol % RSFA | | 88.7 | 84.8 | 87.5 | |
| Total Water, lbs/Ton RS | | 78.3 | 97.7 | 101.1 | |
| Calc. Dry Vent Gas, SCF/Ton RS | | 7291 | 7260 | 7476 | |
| Mineral $CO_2$ Decomposed, % | | 43.5 | 45.8 | 40.9 | |
| MATERIAL BALANCES: | | | | | |
| Ash, wt % (measured) | | 100.2 | 100.3 | 98.0 | |
| Basis for Yields & Mat'l. Bal. | | SS | SS | SS | |
| Overall Balance, wt % | | 99.6 | 100.4 | 102.1 | |
| Organic Carbon Balance, wt % | | 98.0 | 99.7 | 101.7 | |
| Total Carbon Balance, wt % | | 98.5 | 99.7 | 101.2 | |
| Organic Hydrogen Balance, wt % | | 95.3 | 99.3 | 97.2 | |
| Water Balance, wt % | | 149.9 | 158.1 | 192.1 | |
| Gas Loss, SCF/Ton RS (dry) | | 5554 | 5503 | 5946 | |
| HEAT BALANCE: | | | | | |
| Heat of Combustion, MBtu/Ton RS | | 464.6 | 488.5 | 502.4 | |
| Unaccounted Heat, MBtu/Ton RS | | 83.6 | 44.1 | 74.4 | |
| SHALE OIL PROPERTIES: | | | | | |
| Gravity, °API | | 20.2 | 19.9 | 20.0 | |
| Ramsbottom Carbon, wt % | | 1.95 | 1.94 | 2.06 | |
| Ash, wt % | | 0.01 | 0.02 | 0.01 | |
| GAS PROPERTIES (DRY): | | | | | |
| Moisture, lbs/MSCF of dry gas | | 3.8 | 7.1 | 7.2 | |
| Gross Heating Value, Btu/SCF | | 102.6 | 118.1 | [illegible] | |
| $O_2$ vol % | | 0.4 | 0.4 | 0.0 | |
| $CO_2$ vol % | | 27.0 | 26.5 | 26.4 | |
| SPENT SHALE: | | | | | |
| Fischer Assay, Gal/Ton SS | | 0.0 | 0.0 | 0.0 | |
| Organic Carbon, wt % | | 1.73 | 2.09 | 2.17 | |

(1) Retort No. 1 runs - no prefix; Retort No. 2 runs - "B" prefix.

(2) Measure Recycle + Dilution Gas Rate Minus Wet Gas Loss

OSRC-11

REVISED SPENT SHALE ANALYSIS & L.P. CALC.

4-13-65 RH

Oil Shale
FRC-JND

May 21, 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 April 10 to May 15, 1965)

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

Yours truly,

R. H. Cramer
Program Manager

RHC:rl
Attachments

cc: W. L. Jensen