

A LABORATORY STUDY OF
CO-CURRENT AND REVERSE-
CURRENT BATCH RETORTING
OF COLORADO OIL SHALE

by

ROBERT E. CRUMB

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A Thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science in Petroleum Engineering.

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CONTENTS

	<u>Page</u>
ABSTRACT.....	vii
ACKNOWLEDGMENTS.....	x
INTRODUCTION.....	1
Background Information.....	2
Purpose and Scope.....	5
MECHANICS OF THERMAL DEGRADATION.....	8
PRINCIPAL METHODS OF RETORTING COLORADO OIL SHALE....	16
The Bureau of Mines Method.....	17
The Union Oil Company Method.....	22
The Oil Shale Corporation Method.....	25
The Reverse-Current Method.....	28
LABORATORY BATCH RETORTING.....	32
Equipment.....	33
Procedures.....	39
CO-CURRENT BATCH RETORTING.....	41
Runs.....	41
Results.....	42

	<u>Page</u>
REVERSE-CURRENT BATCH RETORTING.....	49
Runs.....	49
Results.....	50
ANALYSIS OF RAW FEED-SHALE AND SPENT SHALE.....	57
DISTILLATION OF CRUDE OIL SAMPLES.....	61
CONCLUSIONS.....	65
APPENDIX.....	68
SELECTED BIBLIOGRAPHY.....	82

ILLUSTRATIONS

	<u>Page</u>
Figure 1. The Bureau of Mines Method.....	21
2. The Union Oil Company Method.....	24
3. The Oil Shale Corporation Method.....	25
4. Comparison of Co-Current with Reverse- Current Retorting.....	31
5. Laboratory Setup.....	36
6. Flow Diagram.....	37
7. Batch Cross-Section.....	38
8. Laboratory Runs vs. NTU Runs.....	47
9. Laboratory Runs vs. Gas-Combustion Runs..	48
10. Laboratory Runs (Yields) vs. Marathon Reverse-Current Runs.....	55
11. Laboratory Runs (°API) vs. Marathon Reverse-Current Runs.....	56
12. Raw Feed-Shale Sieve Analysis.....	59
13. Crude Oil Distillations.....	62

TABLES

	<u>Page</u>
Table 1. Co-Current Summary.....	45
2. Co-Current Material Balance.....	46
3. Reverse-Current Summary.....	53
4. Reverse-Current Material Balance.....	54
5. Raw Feed-Shale Sieve Analysis.....	60
6. Crude Oil Distillations.....	63
7. Run A#1 (Co-Current).....	69
8. Run A#2 (Co-Current).....	70
9. Run A#3 (Co-Current).....	71
10. Run A#4 (Co-Current).....	72
11. Run B#2 (Reverse-Current).....	74
12. Run B#3 (Reverse-Current).....	76
13. Run B#4 (Reverse-Current).....	78
14. Run B#6 (Reverse-Current).....	80

ABSTRACT

The magnitude of oil reserves in the oil shale area of Colorado, Utah, and Wyoming is staggering. These reserves are several times the total known crude oil reserve of the entire world.

The oil in this area is contained within a generally homogeneous deposit called the Green River formation, which is a fine-grained marlstone impregnated with approximately 13.8 percent by weight organic matter called "Kerogen". Kerogen is a rubbery solid, intermediate between coal and petroleum, that is essentially insoluble in normal petroleum solvents. Essentially, the only practical manner of recovering kerogen is by physically changing it by pyrolysis or thermal degradation to form a condensable vapor.

Thermal degradation of kerogen is a complex process involving alteration steps in which kerogen is changed to bitumen and then to a condensable vapor. The process varies with the rate of application of heat and with the characteristics of the retort used. Gas channeling during retorting causes poor recovery.

Four retorting methods that appear to have commercial potential for retorting Colorado oil shale are discussed: (1) The Bureau of Mines Method, (2) The Union Oil Company Method, (3) The Oil Shale Corporation Method, and (4) The Reverse-Current Method.

A working model batch retort used in this investigation is described. Fifteen runs were made on the retort, but due to modifications made during seven of the runs, only eight runs are reported: four co-current and four reverse-current.

Nineteen characteristics or properties of each run are summarized in tabular form. Material balance calculations made for each run are reported in tabular form. Time summaries for each run are given in the Appendix.

Data from these runs are spot checked against published data for similar experiments. Major differences do occur in spot-checking reverse-current retorting data from this investigation with published results (extrapolated) from other reverse-current experiments.

A.S.T.M. distillations of co-current and reverse-current composite crude-oil samples from this investigation are recorded, and a sieve analysis of the raw feed-shale used in this investigation is given.

Many differences are noted when comparing co-current with reverse-current runs in this investigation. Large volumes of ammonia water were obtained for both co-current and reverse-current runs.

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INTRODUCTION

During the past two or three years there has been a revived interest in the enormous deposits of oil shale lying in Colorado, Utah, and Wyoming. There was an earlier oil shale "boom" during the early twenties (1920's). Newspapers in oil shale area of Colorado, Utah and Wyoming (hereinafter referred to as "the oil shale area") have acclaimed the recent interest to be the forerunner to an economic boom that will make the oil shale area the largest oil field in the world.

This renewed interest prompted the writer to build a bench-model batch retort to compare retorting characteristics of co-current retorting with those of reverse-current retorting. According to the literature, reverse-current retorting is better than

co-current retorting in certain respects. However, this investigation showed the opposite to be true for the same set of conditions considered.

Pertinent literature with respect to oil shales and the purpose and scope of this investigation are discussed under: (1) Background Information, and (2) Purpose and Scope of Thesis.

Background Information

The magnitude of oil reserves in the oil shale area is staggering. Thorne and others (1964) report that in Colorado there are 400 billion barrels of oil in place in oil shale sections 15 to 2,000 ft thick, which average 15 gal of oil per ton and underlie 1,380 square miles; in Utah there are 120 billion barrels of oil in place in oil shale sections 15 to 150 ft thick, which average 25 gal of oil per ton and underlie 5,400 square miles; and, in Wyoming there are 12 billion barrels of oil in place in oil shale sections 15 to 80 ft thick, which underlie 9,600 square miles. Stockmar (1964) points out that the oil content of the oil shale deposits lying in the oil shale

area is several times the total known crude oil reserve of the entire world and up to 30 times the known United States reserves.

Although the Colorado, Utah, and Wyoming oil shale deposits are probably the most important in the world, Thorne and others (1964) report that widely distributed oil shales occur in many countries in sedimentary formations of many geologic ages. For example, Sweden, Germany, Scotland, France, China, and Brazil are countries that have oil shale industries of varying degrees of importance.

The abundance of oil shale around the world has challenged man for more than 100 years about "ways to obtain oil from the rock." Consequently, as Thorne and others (1964) report, over 2,000 patents have been granted for various oil shale retorting or beneficiation techniques. They emphasize that in spite of the large number of patents that have been granted, only a few retorting systems have been used industrially, and no single process seems best for all types of shales or economic conditions.

The oil shale deposits in the oil shale area are contained within a generally homogeneous sedimentary

deposit called the Green River formation. The Green River formation is a fine-grained marlstone impregnated with organic matter called "kerogen." A 25-gal-oil-per-ton sample from the Mahogany zone of the Green River formation as reported by Thorne and others (1964, p. 7) contains:

(1) 13.8 percent by weight organic matter which consists

of:

(A) Carbon.....	80.5 percent
(B) Hydrogen.....	10.3 percent
(C) Nitrogen.....	2.4 percent
(D) Sulfur.....	1.0 percent
(E) Oxygen.....	<u>5.8 percent</u>
	100.0 percent

(2) 86.2 percent by weight mineral matter which consists

of:

(A) Carbonates (principally dolomite).....	48 percent
(B) Feldspars.....	21 percent
(C) Quartz.....	13 percent
(D) Clays (principally illite).....	13 percent
(E) Analcite.....	4 percent
(F) Pyrite.....	<u>1 percent</u>
	100 percent

Allred (1964, p. 47) states that the organic matter called kerogen is a (rubbery) solid essentially insoluble in petroleum solvents and it is intimately mixed with the mineral grains in the rock. The kerogen molecule (there is some uncertainty about calling the minute particles of organic matter molecules) differs from petroleum mainly in geometry. Typical petroleum molecules are linear chains with some rings and branches but with little linking between chains. Kerogen, on the other hand, has complex cross linking. Hawley (1929) reports that there is approximately two percent soluble organic matter (natural bitumen) mixed with kerogen.

Purpose and Scope

This investigation was undertaken, first, to design and build a working-model batch retort of bench-top-size, and second, to compare the characteristics of co-current with reverse-current retorting.

The batch retort in this investigation consists of two insulated cans each holding 3,600 g of raw feed shale, a preheating-furnace system, a switching-manifold for

changing the direction of the gas stream to and from the batches, and a condensing system.

Fifteen runs were made on the retorting system. However, during seven of the runs, modifications made on the equipment prevented a material balance check, so only eight runs are reported in this investigation. Four co-current and four reverse-current runs are reported.

No attempt is made to optimize over a range of operating conditions. Nevertheless, data from these runs are believed to be meaningful when spot-checked against published data for similar retorting conditions. Major differences which are discussed later do occur in spot-checking reverse-current retorting data from this investigation with published results from other reverse-current experiments.

Nineteen characteristics or properties of each run are summarized in tabular form for comparisons. In addition, a material balance (vent gas molecular-weight calculation) is made for each run; A.S.T.M. distillations of co-current and reverse-current composite crude oil samples are plotted; and, a sieve analysis of the raw

feed-shale is presented.

Two sections of this investigation are a review of literature regarding oil shale: (1) Mechanics of Thermal Degradation (a review emphasizing the physical changes undergone by kerogen during thermal degradation) and (2) Principal Methods of Retorting (a review describing retorting methods that have shown a good potential for use in the oil shale area).

MECHANICS OF THERMAL DEGRADATION

Many different methods have been tried to extract organic matter, or kerogen, from oil shale. Most of them have been unsuccessful. For example, all suggested methods of leaching with solvents have been unsuccessful because of the insolubility of kerogen in normal petroleum solvents. The only successful methods of extracting kerogen from oil shale rely upon physically changing the kerogen by heating it to essentially 900°F in order to form a condensable vapor. These methods are called "thermal degradation" or "pyrolysis."

Guthrie (1964) generalizes the various techniques of heating to give pyrolysis into the following categories:

- (1) Indirect heating through the wall of the retorting vessel.

- (2) Direct heating by hot gases from combustion within the retorting vessel.
- (3) Heat transfer from an externally heated carrier fluid.
- (4) Heat transfer from recycled hot solids.

Each of these techniques has been used successfully in a particular application that best suits that technique; but, what works for retorting one kind of shale may not work for another.

Thermal degradation of kerogen is not a simple process; it is a complex chain of events. Guthrie (1938) reports that kerogen is not vaporized or rendered soluble upon the application of heat, even under reduced pressure, but that it is first converted to bitumens after which several consecutive processes are simultaneously involved. Williamson (1964), in his review of the literature, summarizes the steps that take place during thermal degradation, each step requiring a correspondingly higher temperature than the preceding step:

1. Water and gases are evolved.
2. Kerogen is converted to a bitumen.
3. The bitumen is altered.

4. The bitumen is disassociated to oils, gases, and compounds (with oxygen and nitrogen).
5. The oils are vaporized.
6. Fixed carbon is burned, and both organic residue and inorganic compounds (carbonates) are decomposed.

Probably the best generalized information about what happens to kerogen during thermal degradation has been given by Guthrie (1938, p. 108). He listed the following relationships at various temperatures:

325°C (620°F) -- Kerogen softens, swells, becomes black, has general appearance of soft bitumen. Solid nitrogen compounds are converted to soluble compounds.

300 to 405°C (570 to 760°F) -- The "water-producing" period -- water of combination and ammonia are released. Easily volatilized organic nitrogen compounds and hydrogen sulfide are formed.

390°C (740°F) -- Heavy oil vapors and gas to be formed are distilled.

500 to 600°C (930 to 1,110°F) -- Production of condensable oil vapors ceases.

+600°C (+1,110°F) -- Noncondensable hydrocarbons and ammonia continue to be produced.

At elevated temperatures, the steps generally follow each other closely. In a retort, however, the steps occur simultaneously because of various stages of decomposition

and different temperatures in different parts of the retort.

Each of the thermal degradation steps are altered in different retorting systems because of varying conditions inherent to each system; hence the products of retorting are often much different. In Colorado oil shale the best retorting systems have converted about 66 percent of the organic material to oils, 9 percent to gases, and 25 percent to coke. The coke is usually burned to provide heat for retorting.

Karrick (1923) reports that the amount and quality of oil that can be produced from oil shale may be made to vary to an important degree by changing the temperature relations existing in the shale charge during thermal degradation. Variations in yield and quality of oil produced are very largely the result of the rate of heat application during retorting, and the extent to which thermal degradation has progressed. Rapid heating gives large yields of crude oil having a low API gravity (15 to 20°API), high pour point (around 90°F), small percentage of light distillate, high carbon residue, and large

amounts of dust and carbon particles in suspension. On the other hand, slower heating gives a lower yield of oil than obtained at higher rates; but the oil has a higher API gravity (20 to 30°F), lower pour point (60 to 80°F), higher percentage of light distillate, lower carbon residue, and low amounts of suspended dust and carbon particles. Karrick (1923) reports that for very slow thermal degradation (gradual increase in temperature to 1,110°F over 24 hours) the product oil from condensed gases is clear and transparent, bright green, and fluorescent.

In commercial retorting systems it is generally believed impractical to try to obtain the light, bright green oil described by Karrick (1923) because of the time element involved. The poor heat conducting properties of the shale makes it impossible to have rapid propagation of heat without the use of high temperatures which are detrimental to the quality of the crude obtained from thermal degradation.

Shale oil from most retorting procedures is a black, waxy mixture of 15 to 20°API gravity with a high pour point

of about 95°F. Thorne and others (1964) report that utilization of shale oil as a feedstock for refining presents special problems because its composition differs in some respects from that of petroleum, and because shale oils produced by different processes differ from each other. The oil is quite viscous and is high in sulfur and nitrogen content. Carver (1964) reports, however, that after treatment (hydrogenation) shale oil is as good a feedstock for refining as other crude oil.

Thorne and others (1964) report that shale oil contains large quantities of olefinic hydrocarbons. In this respect it resembles the products from thermal cracking of petroleum. In general, shale oil contains larger quantities of nonhydrocarbons, particularly nitrogen and oxygen compounds, than does petroleum. Shale oil may be considered intermediate between petroleum and coal tar with respect to the degree of unsaturation.

Allred and Nielson (1965) have done considerable work in establishing temperature and time relationships with respect to the thermal degradation of oil shale. Their work shows that a temperature range of 900 to 1,000°F

is most desirable for optimum yield and product. Their work also shows that a higher API gravity oil occurs close to the point of optimum yield than occurs on either side. Data from Allred and Nielson (1965) show that even for relatively small shale particles, several minutes of heating at temperatures of 900 to 1,000°F are required to give satisfactory separation of oil from shale.

Usually the time required for retorting is much longer than a few minutes, as Savage (1966, p. 20) states, "The Bureau of Mines personnel reported that at a temperature of 850°F, 90 percent of the retorting can be accomplished in less than five minutes, but in a commercial retort it sometimes takes hours to complete retorting."

Savage (1966) believes that the long time usually required for retorting is caused by poor heat transfer because of dead spaces in the retort. The dead spaces are formed by interlocking particles of broken shale descending as a mass through a retort. He stated that there is good contact between particles of shale and hot gas in the vicinity of the walls of the retort but that in the balance of the retort the shale flows as a mass

through which the gas cannot circulate uniformly.

The gas follows the path of least resistance and it channels near the walls of the retort, where shale undergoes thermal degradation quite rapidly. Elsewhere in the retort, however, there are dead spaces where shale undergoes thermal degradation rather slowly.

PRINCIPAL METHODS OF RETORTING

Of the more than 2,000 patents that have been granted for methods of retorting oil shale, at this time only four appear to have commercial potential for retorting Colorado oil shale. These are (1) The Bureau of Mines Method, (2) The Union Oil Company Method, (3) The Oil Shale Corporation Method, and (4) The Reverse-Current Method.

Each of these methods differs from the other in (1) the mechanics of heating the oil shale to about 900°F, in (2) the mechanics of condensing the educt gas formed during thermal degradation of the oil shale, and (3) in the mechanics of disposing of the spent shale after retorting. In the Reverse-Current Method, considerable thought has been given to the possibility of retorting

the oil shale "in-situ," i.e., in place in the ground.

The Bureau of Mines Method

The Bureau of Mines operated an oil shale demonstration plant near Rifle, Colorado, from 1949 to 1956, after which the program was discontinued. Its research consisted of mining and retorting the oil shale, and of refining the produced oil. Several retorting systems were investigated before the "Bureau of Mines Method" or "the gas-combustion" method as it is sometimes called, evolved from the planning stage to a demonstration plant.

The Bureau's plant utilizing the "gas-combustion method," meets many requirements that apply specifically to the Rifle oil shale area. The plant should meet the following requirements for retorting according to Guthrie (1964, p. 8):

1. It should be continuous.
2. It should be capable of high oil recovery efficiency.
3. It must have a high shale throughput rate.
4. It must be thermally self-sufficient; that is, all heat requirements should be supplied from the hydrocarbon gases and/of the residual

carbon produced from the pyrolysis of the organic matter in the shale, rather than from the oil.

5. It should require low capital investment; be mechanically simple; easily operable; and possess a high operating factor with low capital costs.
6. It should lend itself to enlargement into high tonnage retorts rather than a multiplicity of small units.
7. It should require little or no water as the Colorado deposits are located in semi-arid regions, where the only water available is costly.
8. It should be capable of efficiently processing oil shale of as wide a particle-size range as possible to minimize the amount of crushing, screening, and handling which otherwise would be required.

In the Bureau of Mines method, raw feed-shale crushed to -3 in. to +1/2 in. size enters the top of a simple vertical gravity-fed fire-brick-lined cylindrical shaft, in which raw feed-shale is fed downward and hot recycle and combustion gases are passed upwards. The descending bed of raw feed-shale successively passes through preheating, retorting, combustion, and cooling stages. Recycle gas is injected at the bottom of the retort to absorb heat from the hot spent shale coming from the combustion zone.

Air is injected at the combustion zone to provide oxygen to burn the heated recycle gas and to burn some of the residual carbon left in the shale from thermal degradation of the kerogen in the retorting zone.

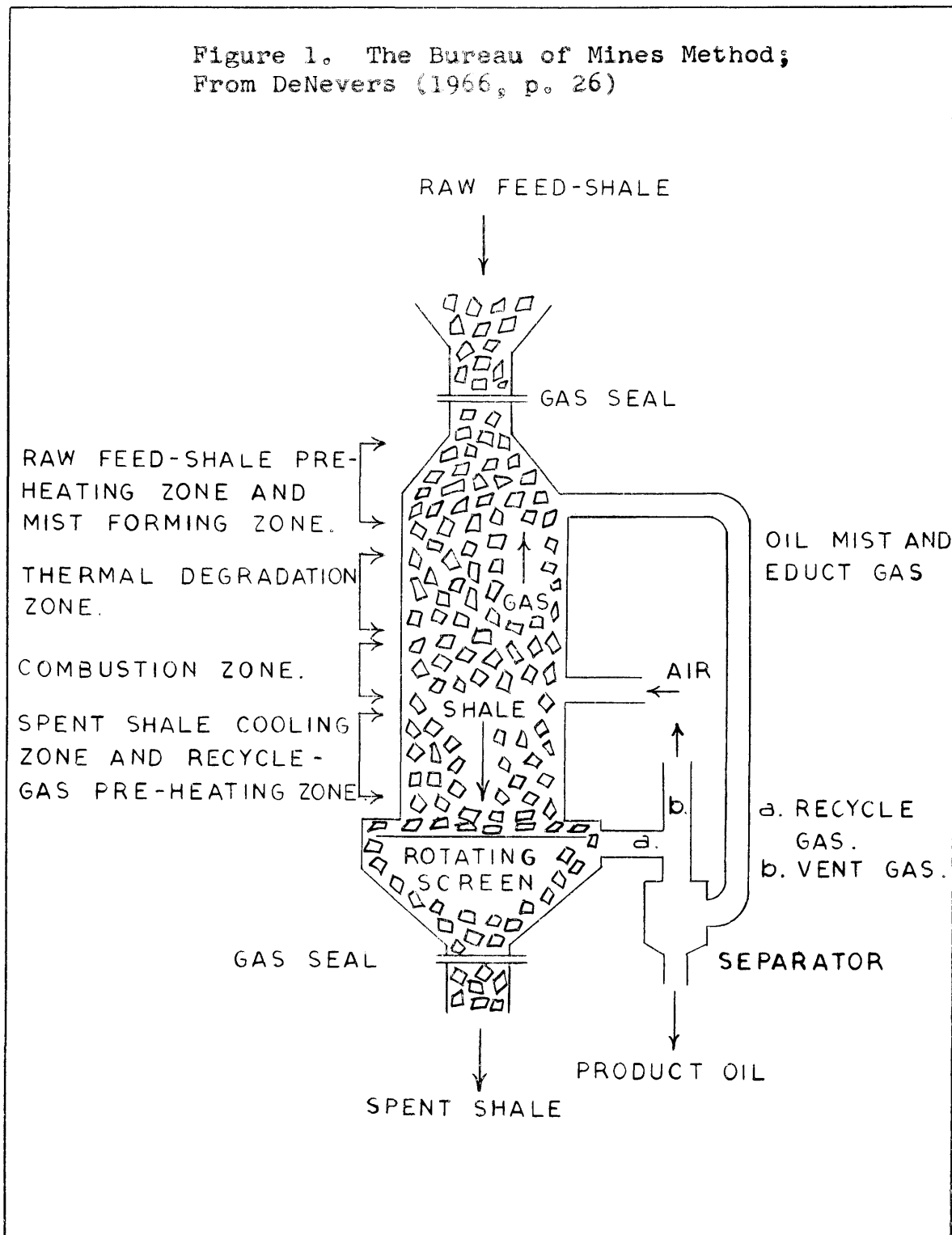
The resulting hot gases from the combustion zone travel upward through unburned shale above the combustion zone and heat the unburned shale to retorting temperatures (about 900°F) during which time thermal degradation of the kerogen in the oil shale takes place. The oil and gaseous products from the retorting zone are swept along with combustion gases into the cooler, incoming raw feed-shale above the retorting zone. In the cooler shale zone (raw feed-shale preheating zone), the oil and gaseous products from the retorting zone are condensed into a mist which is carried out of the retort with the total gas stream to condensing systems.

External condensing systems separate shale oil from the gas stream and divide the gas stream. Part of the gas stream is recycled to the base of the retort to be used as cooling gas and part is piped elsewhere for use as product gas.

The Oil and Gas Journal (1964, p. 69) states that the operational characteristics of the Bureau of Mines retort varied with throughput rate. At a capacity of 6 tons per day, it yielded 98 percent of Fischer assay. The yield decreased to 92 percent of Fischer assay when retorting rates were increased to 20 tons per day, and was only 70 percent of Fischer assay when retorting rates were increased to 150 tons per day on the same equipment. The drop in efficiency was believed to be due to poor heat transfer characteristics caused by channeling of retorting gases in the shale bed.

A schematic diagram of the Bureau of Mines method is shown in Figure 1, page 21. Each of the major components is identified.

Figure 1. The Bureau of Mines Method;
From DeNevers (1966, p. 26)



The Union Oil Company Method

The Union Oil Company of California operated a pilot plant in Colorado between 1955 and 1958, during which time the company developed a retorting method operating on a downdraft basis with a large and unique swinging piston called a "rock pump" at the base of the retort. The rock pump delivers raw feed-shale of the same size used in the Bureau of Mines method, into the retort from an external supply hopper.

The rock pump cycle consists of four steps:

- (1) Downstroke (filling with a charge of raw feed-shale from the hopper -- step "b" in Figure 2, page 24).
- (2) Rotation (charge of raw feed-shale is rotated from beneath the hopper to beneath the retort).
- (3) Upstroke (charge of raw feed-shale is pushed upward into the retort -- step "a" in Figure 2, 24).
- (4) Rotation (discharged piston assembly is rotated back from beneath the retort to beneath the hopper).

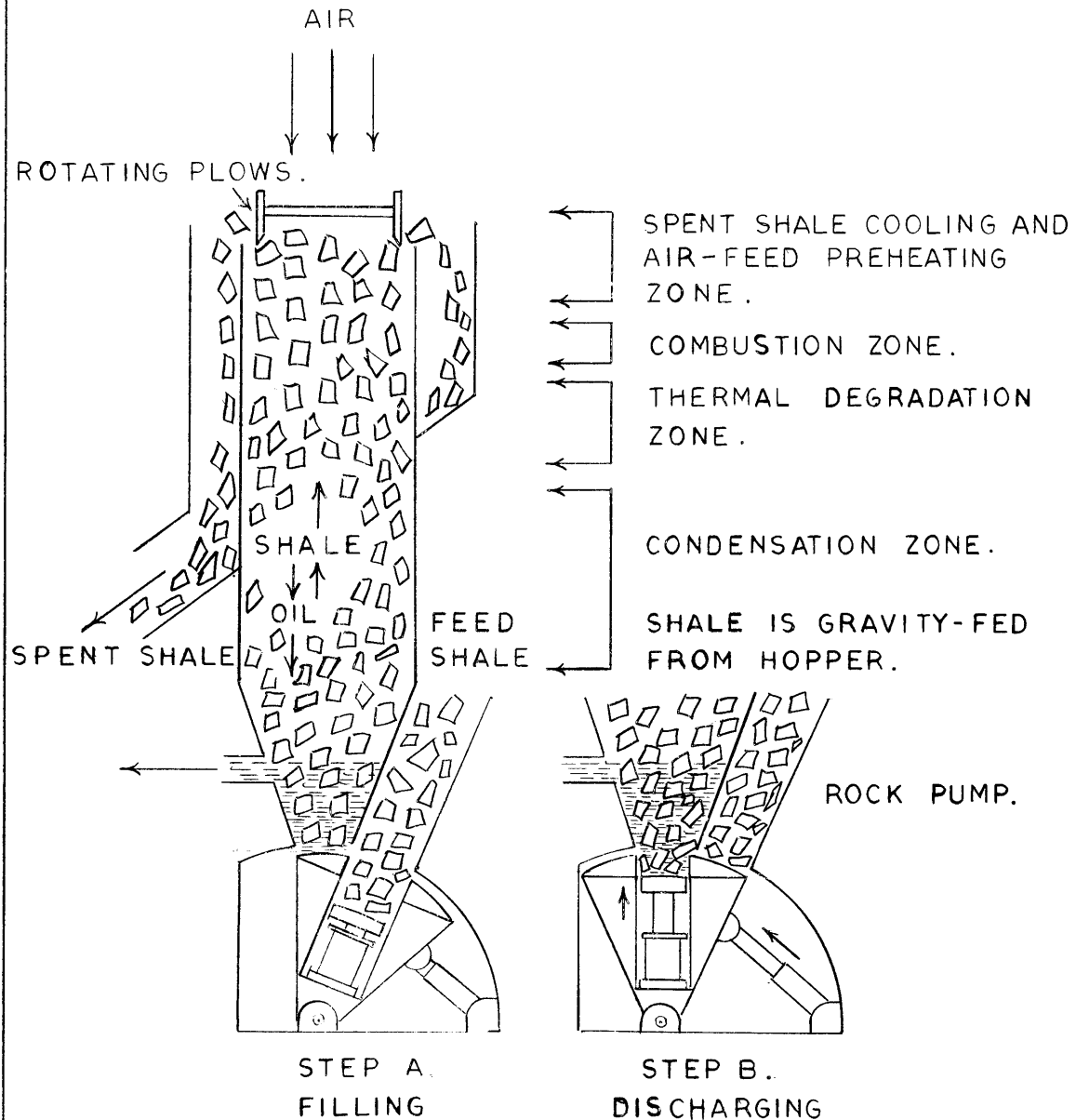
Thorne and others (1964) report that air enters the top of the retort and combustion takes place about two feet below the top of the shale bed. Hot combustion gases from

the burning zone travel downward through unburned shale below the combustion zone, heating the unburned shale to retorting temperatures (about 900°F), during which time thermal degradation of the oil shale takes place. The oil and gases produced in the retorting zone are condensed on the incoming cold raw feed-shale from the rock pump at the bottom of the retort. Oil is withdrawn from the bottom of the retort as a liquid; gas is withdrawn with the oil and separated for plant use.

Spent shale leaves the retort at the top where incoming air cools the spent shale, or ash, to some extent. Rotating plows at the top of the retort break up spent shale that may have clinkered; broken spent shale falls off the top of the retort onto discharge chutes.

It is generally understood that the Union Oil Company retort has achieved a retorting rate of 1,200 tons of shale per day through use of automatic controls. It is also generally understood that Union Oil Company has considered making a retort of a similar design that could handle 3,000 tons of shale per day.

Figure 2. The Union Oil Company of California Method; From DeNevers (1965, p. 26).



The Oil Shale Corporation Method

The Oil Shale Corporation method differs from the Bureau of Mines and the Union Oil Company methods in one particular respect; that is, combustion and decomposition are separated in the Oil Shale Corporation method; they are not in the other processes.

The Oil Shale Corporation method consists mainly of a modification of the Aspeco process which combines retorting and simultaneous grinding of the raw feed-shale by solid-to-solid contact with hot ceramic grinding balls of 1-in. diameter. These balls are pre-heated to 1,110°F to provide heat for retorting as indicated in Figure 3, page 27, which illustrates the steps of the process.

DeNevers (1966, p. 26) states that the crushed shale (crushed to -1/2 in.) is fed into a primary rotating horizontal kiln along with heated ceramic balls. As the kiln turns, the balls pulverize and heat the shale; the kerogen is decomposed and the oil is driving off as vapor. The partially cooled balls and the retorted shale are drawn off at separate ends of the primary rotary kiln.

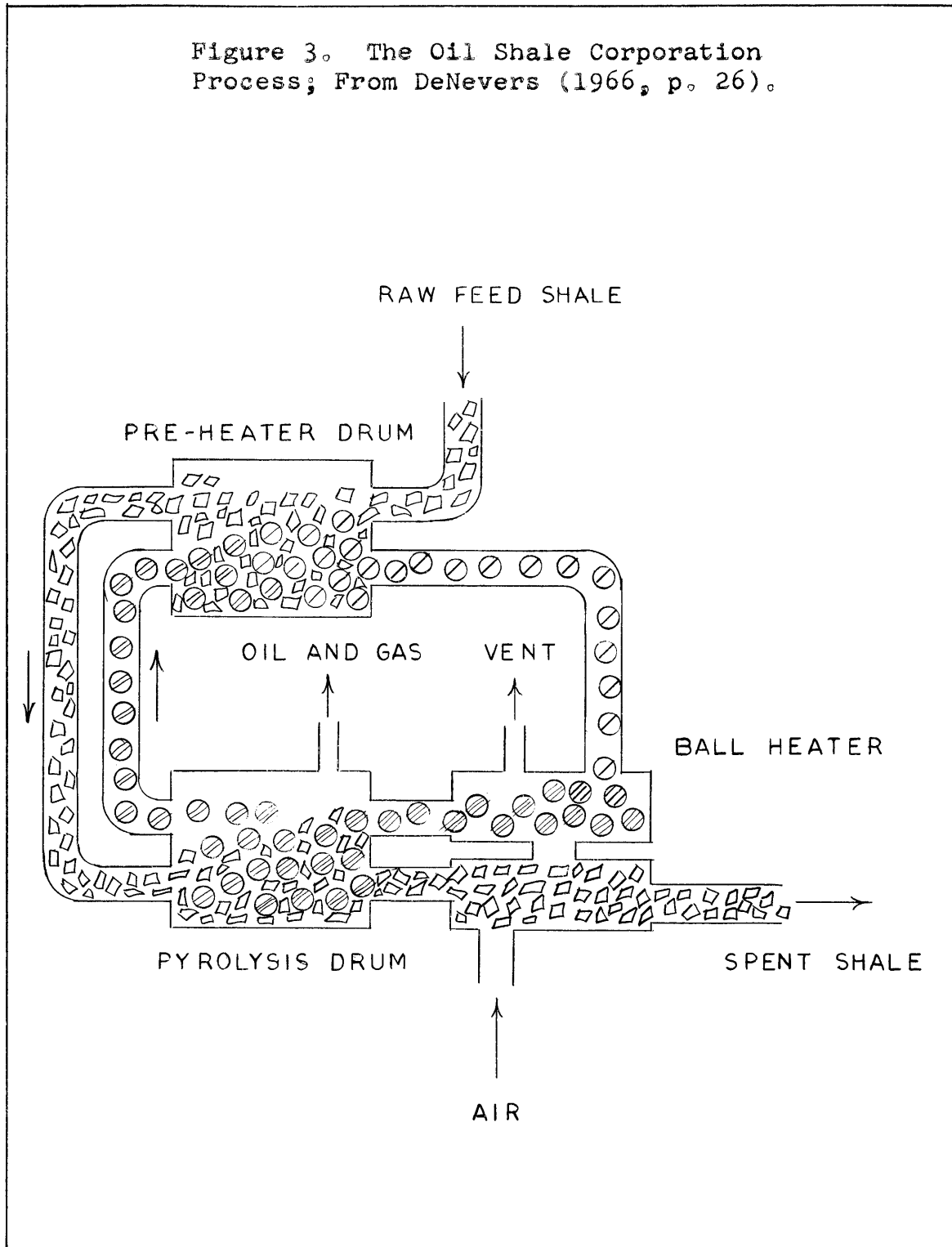
The retorted shale from the primary rotary kiln is

charged into a ball-heating furnace where the residual carbon left on the shale is burned to heat the ceramic balls. The partially cooled balls leaving the primary rotary kiln are routed to a preheating kiln where cold, raw feed-shale is preheated before it is charged into the primary rotary kiln. The oil and gases produced in the primary rotary kiln are drawn off and treated (an unpublished step) and carried to external condensing systems, which separate the shale oil from the product gas.

Part of the product gas is burned in the ball-heating furnace along with residual coke in the spent shale from the primary rotary kiln. Spent shale, or ash, is discharged from the ball-heating furnace.

The Oil Shale Corporation is a relative newcomer to the field of oil shale work; the corporation, known as TOSCO, was founded in 1955. It has hopes of scaling up pilot-plant operations of 150 tons of oil-shale per day capacity, to "semiworks" operations handling 60,000 tons of oil shale per day by the end of 1966. It also has hopes of commercial operations by the end of 1967.

Figure 3. The Oil Shale Corporation Process; From DeNevers (1966, p. 26).



The Reverse-Current Method

The three previously mentioned methods of retorting oil shale require prior mining, crushing and retorting on the surface of the ground, and disposing of the spent shale. Furthermore, oil from the retorting step must be treated before it can be sent to a pipeline for transport to refineries. Reverse-current retorting, on the other hand, may be adapted to retorting oil shale "in-situ," or in place in the ground; and reverse-current retorting may yield a product oil that can be sent to a pipeline without further refining. Marathon Oil Company calls reverse-current retorting "the reverse-combustion process." Reverse-current retorting can also be used for surface retorting, as is done in the other three mentioned methods, but the results are poor as pointed out later in this investigation.

Reverse-current retorting differs from co-current retorting described for the Bureau of Mines method and for the Union Oil Company method in the manner that air (oxygen) is fed to the advancing combustion zone (Figure 4, page 31). In reverse-current retorting, air is fed against the direction of advance of shale or the combustion zone.

whereas in co-current retorting air (oxygen) is fed from behind the advancing combustion zone.

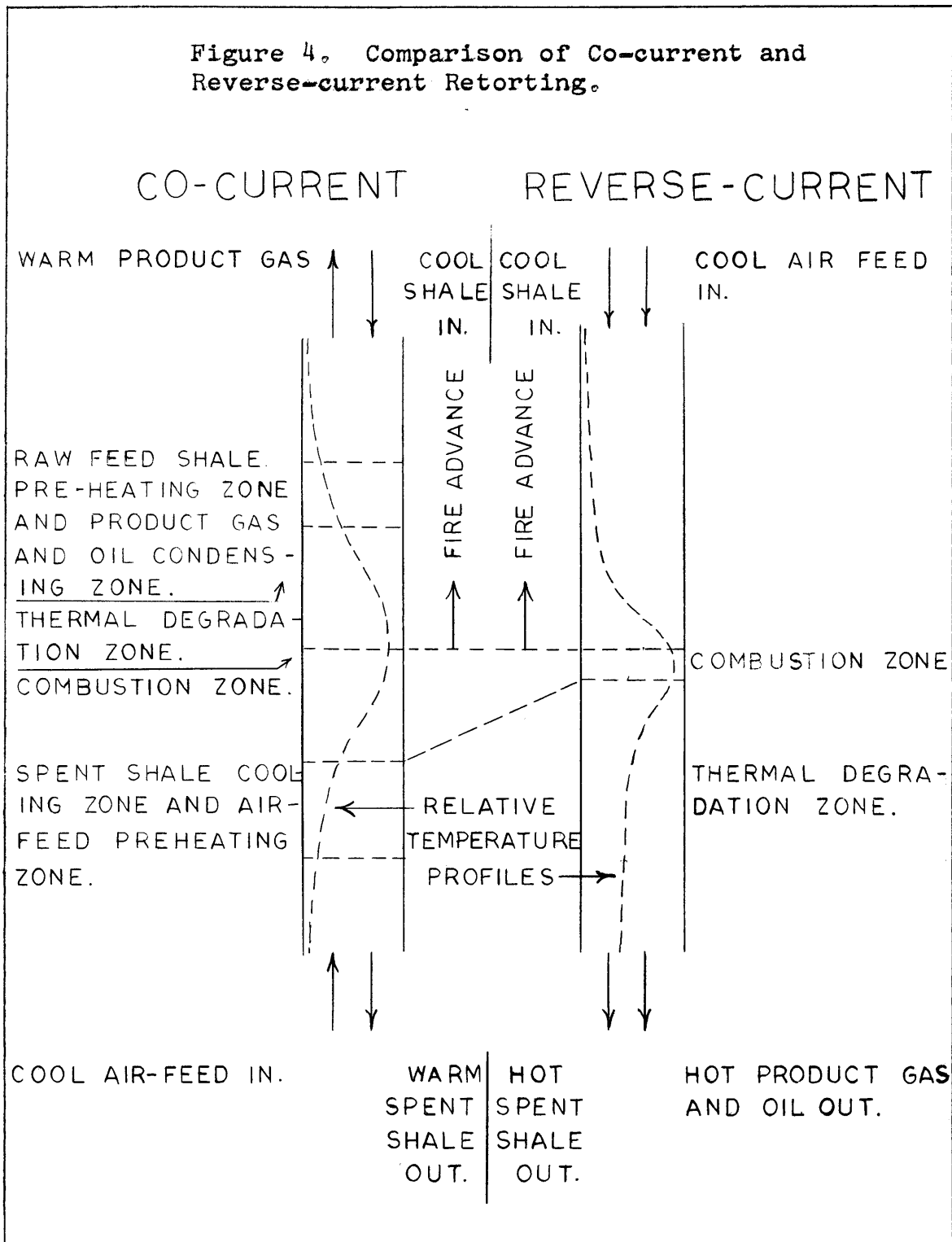
Allred and Nielson (1965, p. 60) state that reverse-current retorting is characterized first by little preheating of the air-feed stream before it enters the combustion zone, second by a sharp temperature rise at the leading edge of the combustion zone, and third by an extensive heated zone to the rear of the combustion zone. The latter zone is where most of the thermal degradation of kerogen in the oil shale takes place.

They explain that reverse-current retorting is possible only if the oxygen consumption at the advancing front is less than the rate at which combustible gases are given off. Heat conducted and radiated ahead of the front and in an opposite direction from the incoming air-feed stream, raises the surface of the unburned raw feed-shale to its ignition temperature. Ignition occurs in combustible gases evolving from the surface of the shale.

Once ignition of surface gases takes place, the surface temperature of the shale rises rapidly and

conducts heat at an increasing rate into the shale particle. In this manner, organic matter within the shale particle is consumed or the oxygen in the air stream is depleted. But, as Allred and Nielson (1965, p. 61) state, heat has already been transmitted to the shale upstream so that it has already reached its ignition point; consequently, if the air (oxygen) rate to the shale is sufficiently low, only a small percentage of the organic matter will be consumed as fuel in keeping the process going. However, this characteristic was not obtained in this investigation; at the pressures used, very large quantities of organic matter in the oil shale were consumed as fuel.

Figure 4. Comparison of Co-current and Reverse-current Retorting.



LABORATORY BATCH RETORTING

One important purpose of this investigation was to design and build functional retorting and condensing systems. The high temperatures reached during retorting (500 to 1,500°F) made it necessary to use special designs for the preheating furnace (a ceramic liner was used), for the batches ("Mono-block" and magnesia insulation were used), and for the switching-manifold (no brass could be used in direct contact with the hot gas stream). The high pour point of the product oil made it necessary to purge all condensers with hot water after each run to collect the maximum amount of the oil condensed during the run. The design and modification period took several months.

Four co-current and four reverse-current runs were made with sufficient data collected for material balance

calculations -- these data and calculations comprise the second purpose of this investigation.

The equipment and procedures for the runs in this investigation are discussed under (1) Equipment, and (2) Procedures.

Equipment

The laboratory equipment used in this investigation is coded and shown on Figures 5, 6, and 7, on pages 36, 37, and 38, respectively. A list of the coded equipment with special remarks follows:

1. Preheating furnace (used to heat manifold and primary batch of shale).
2. Primary batch (batch where thermal degradation of kerogen in oil shale takes place).
3. Auxillary batch (moved to primary batch position for next run when emptying primary batch).
4. Nichrome-steel thermocouple (measures temperature of batch (2)).
5. Cracker (not covered in this investigation).
6. Switching manifold (used to cycle educt gas from burning batch (2) to auxillary batch (3), or from burning batch (2) to cracker (5), or from burning batch (2) to primary condenser (9)).

7. Inlet gas-feed line for reverse-current retorting.
8. Line to primary condenser (9).
9. Primary coil-type condenser (product oil and gases are withdrawn from the bottom of the primary condenser during runs).
10. Secondary condenser.
11. Tertiary condenser.
12. Off-gas line from tertiary condenser (11).
13. Off-gas thermocouple.
14. Primary water trap (used to condense oil vapors that have passed through the tertiary condenser (11)).
15. Secondary water trap (used to condense oil vapors that have passed through the primary water trap (14)).
16. Vent gas meter (measures input air plus product gases).
17. Recycle-gas manifold (this investigation did not cover the effect of recycle gas).
18. Vent gas line.
19. Inlet air-feed line.
20. Inlet air-feed pressure regulator.
21. Recycle gas or propane regulator (only used during preheating in this investigation).
22. Propane tank (for preheating).

23. Recycle gas or auxiliary inlet air-feed compressor (used only as an air-feed compressor in this investigation).
24. Recycle-gas or auxiliary air-feed pressure regulator (used only as an air-feed pressure regulator in this investigation).
25. Air-feed rotameter.
26. Recycle-gas or fuel-gas rotameter (used only for fuel-gas during preheating).
27. Air-feed manometer.
28. Recycle-gas or fuel-gas manometer (used only for fuel-gas during preheating).
29. Air-feed switching manifold (used to change from co-current to reverse-current retorting).
30. Recycle-gas switching manifold (not used in this investigation).
31. Preheating furnace thermocouple.
32. "Mini-mite" temperature indicator.

Figure 5. Laboratory Setup

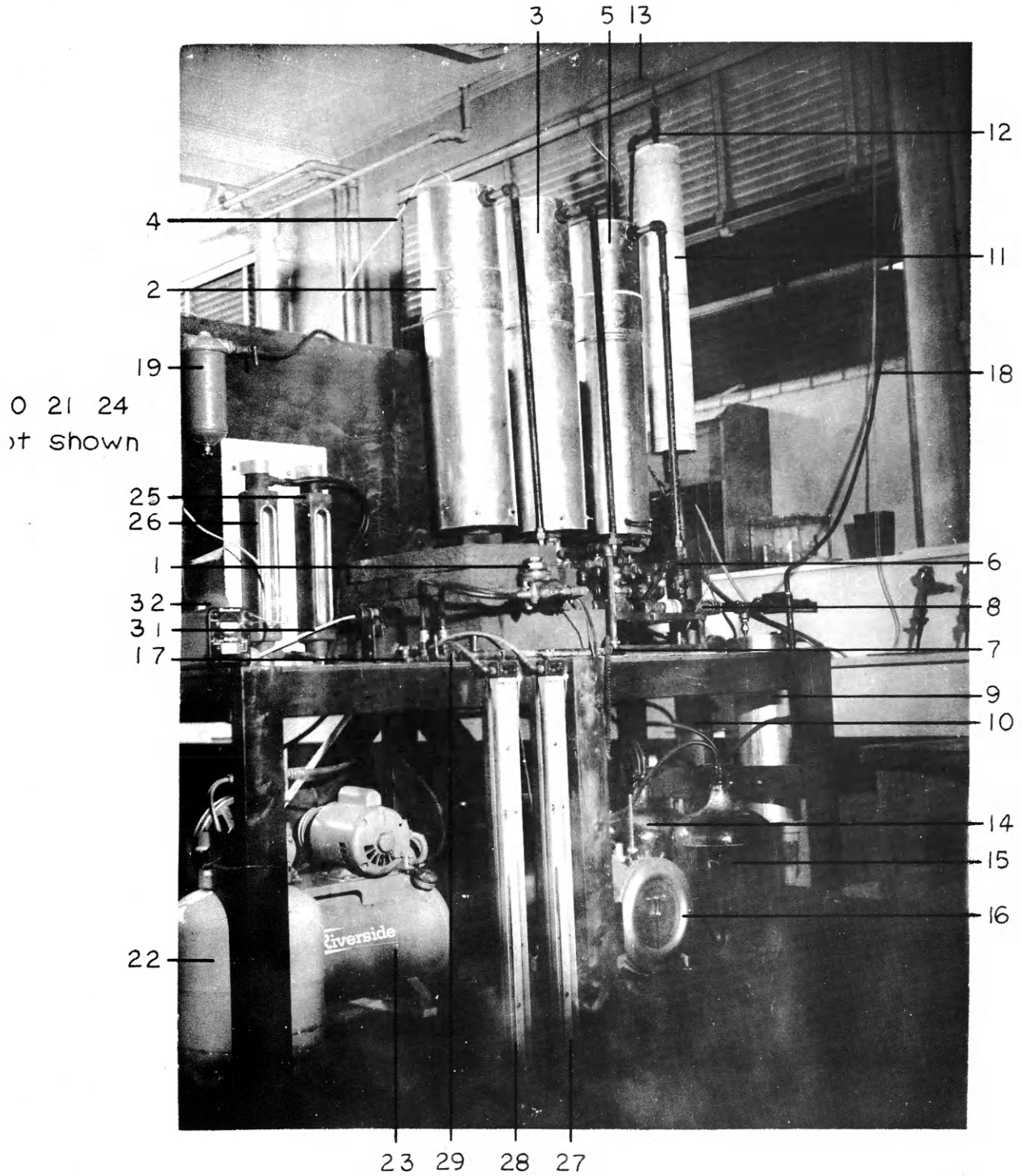


Figure 6. Flow Diagram

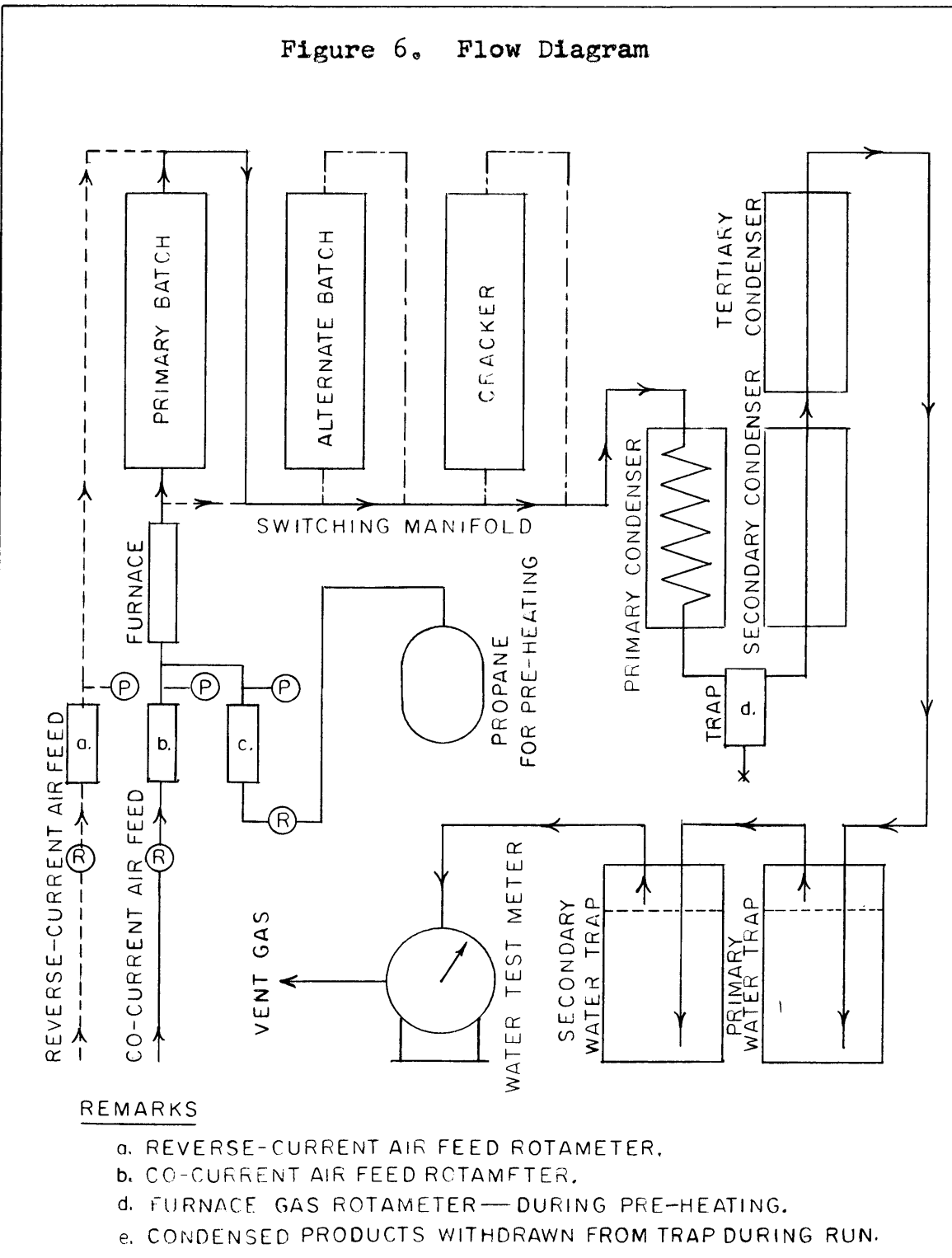
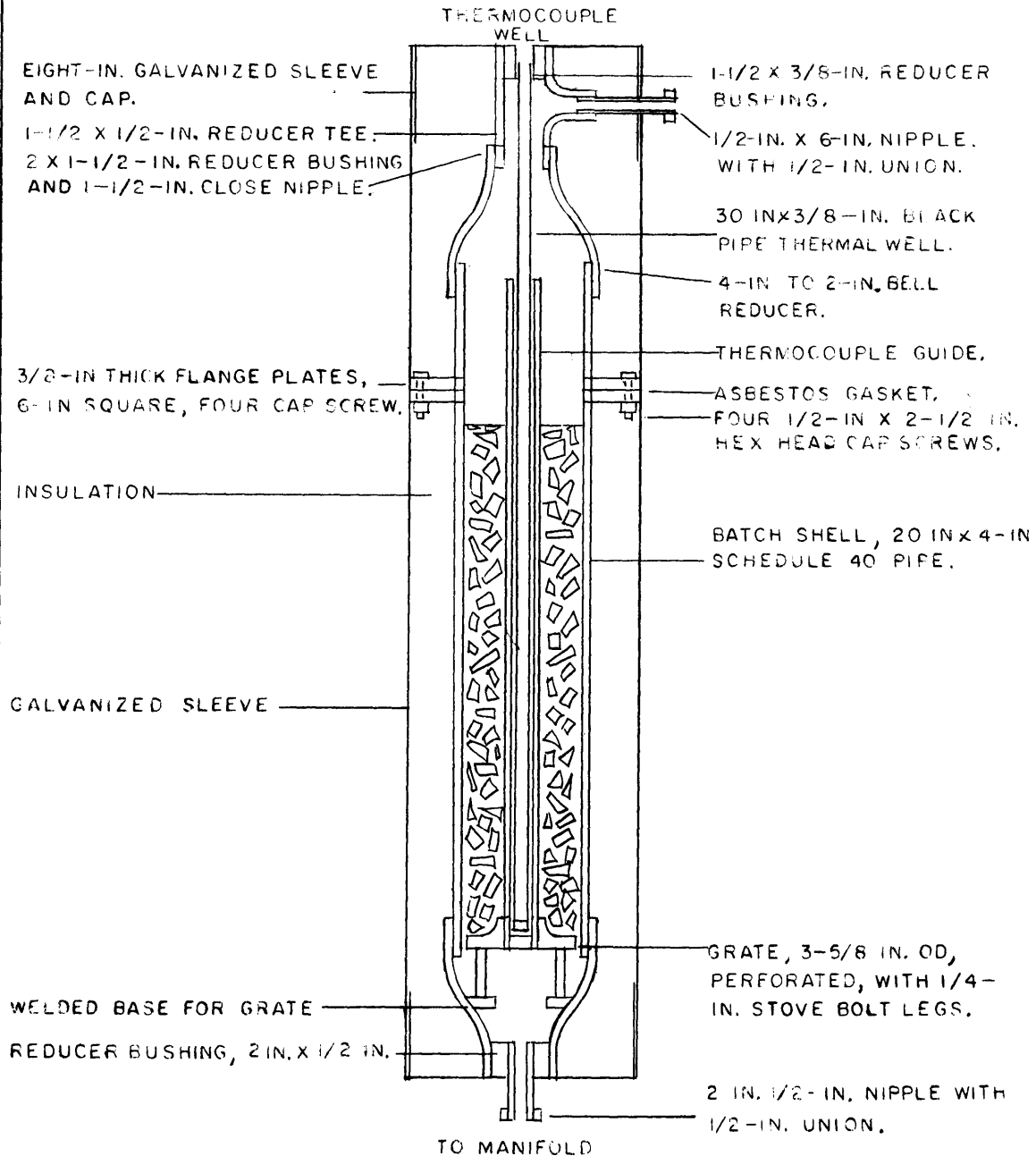


Figure 7. Batch Cross-Section



Procedures

A run consisted of:

- (1) Charging the primary batch with raw feed-shale (approximately 3,600 gms.),
- (2) Mounting the primary batch above the preheating furnace,
- (3) Heating the primary batch with hot combustion gases from the propane pre-heating-furnace until the base of the batch is 500°F, the spontaneous ignition temperature for Colorado oil shale,
- (4) Shutting off propane preheating-furnace and passing air (oxygen) to the batch to obtain spontaneous ignition,
- (5) Maintaining the air-feed rates in the same direction if co-current retorting is desired, or reversing the direction of the air-feed rates and then maintaining conditions if reverse-current retorting is desired, and
- (6) Periodically measuring air-feed rates, batch temperatures at various points, condensing system temperatures, volumes of condensed oil and water, and volumes of total-vent-gas.

The batches were charged by:

- (1) Dismounting the batch from the manifold at the unions,
- (2) Breaking down the batch at the flange,
- (3) Filling the batch with raw feed-shale to the desired height (18 in.),

- (4) Closing the batch and tightening the flange, and
- (5) Remounting the batch on the manifold for the next run. The weight of each batch of raw feed-shale was measured before charging, and the weight of the spent shale was measured after each run when the batch was dismantled.

CO-CURRENT BATCH RETORTING

A co-current run in which hot educt gas from the primary batch was passed directly to the condensing system is shown by a solid line in Figure 6, page 37. Co-current retorting runs in this investigation are discussed under (1) Runs, and (2) Results.

Runs

Four co-current runs are reported in this investigation. Runs A#1, A#2, and A#3 were performed at an air-flux rate of 163.0 standard cubic feet per square foot of retort cross-sectional area per hour, hereinafter referred to as std cu ft/sq ft/hr. Run A#4 was performed at 64.1 std cu ft/sq ft/hr. No difficulties were encountered except those noted on Table 2, page 46.

Results

The results of the four co-current batch retorting runs are tabulated on Tables 1 and 2 on pages 45 and 46 respectively. Tables 7, 8, 9, and 10 on pages 69, 70, 71, and 72, respectively, give a time analysis with special remarks for each run.

Figure 8, page 47, shows a comparison of retorting rates (lb/hr/sq ft) for the laboratory runs with those of the NTU retort (a co-current batch retort operated by the Bureau of Mines) at the same air flux (std cu ft/sq ft/hr). Higher retorting rates are reported for the laboratory runs than are reported for the NTU runs. The lower retorting rates in the NTU retort were probably due to gas channeling.

Figure 9, page 48, shows a comparison of oil yields (percent of Fischer assay), product gas (std cu ft/ton of raw feed-shale), and retorting rates (lb/hr/sq ft of retort cross-section) for the laboratory runs with those of the gas-combustion retort at the same superficial linear gas velocity (ft/sec). It is shown in this comparison that oil yields and product gas yields are

lower, and retorting rates are higher for the laboratory runs than should be expected from extrapolated rates for the gas-combustion runs at the same superficial linear gas velocity (ft/sec).

Significant differences are noted between the quality of oil produced in the laboratory runs and the quality of oil produced by other co-current methods. The laboratory oil is lighter (22.7° compared with 19.0° API); it has a lower pour point (75° compared with 90° F); and it has a lighter color (dark green compared to black) than oils produced by other co-current methods.

Another significant factor noted in this investigation is that at the air-flux rates investigated (82.0 to 163.0 std cu ft/sq ft/hr), high volumes of water are produced. From 40 to 50 percent of the total condensed products for each run is water. This volume of water is four or five times the volumes reported for other co-current methods. The high volumes of water produced in this investigation are probably due to the lower air-flux rates than those of the other co-current methods. The lower air-flux rates would give a correspondingly longer

period of time of retorting at lower temperatures, a condition that would yield water of combination and ammonia (Guthrie, 1938, p. 108). A test for ammonia in water produced during Run A#4 showed a 0.4 normal basic solution.

TABLE 1
CO-CURRENT SUMMARY

ITEM	RUN NUMBER	A#1	A#2	A#3	A#4
1. Fischer Assay, Feed.....	gal/ton	33.3	33.3	33.3	33.3
2. Assay of Spent Shale.....	gal/ton	0.00	0.00	0.00	0.00
3. Percent of Spent to Raw Shale.....		64.9	65.9	64.2	68.2
4. Oil Recovered.....	gal/ton	23.7	23.0	25.5	21.9
5. Oil Rec. Percent Fischer Assay.....		71.1	69.0	76.5	65.8
6. Oil Gravity.....°API @ 60°F		22.7	22.7	22.7	22.7
7. Oil Pour Point.....°F		75	75	75	75
8. Water Condensed.....gal/ton		16.7	16.8	19.3	23.9
9. Percent Water in Condensed Products.....		41.4	42.2	43.0	52.1
10. Maximum Retort Temperature °F.....		980	1,062	945	1,020
11. Air Rate..... std cu ft/min		.212	.212	.212	.144
12. Superficial Gas Velocity.....ft/hr		113.9	113.9	113.9	64.1
13. Air Flux.....std cu ft/sq ft/hr		163.0	163.0	163.0	82.0
14. Air Flux.....std cu ft/ton		5,570	5,450	4,770	5,100
15. Gas Yield.....std cu ft/ton		6,010	5,550	4,420	4,350
16. Firing Time.....hr		1.73	1.72	1.52	3.00
17. Length of Run.....hr		2.60	2.78	2.17	4.00
18. Rate of Retorting.....ft/hr		.87	.87	.99	.50
19. Rate of Retorting.....lb/hr/sq ft		58.7	59.7	106.3	34.0

TABLE 2

CO-CURRENT MATERIAL BALANCE

ITEM	RUN NUMBER	A#1	A#2	A#3	A#4
1. Raw Feed Shaleg.	3,591	3,632	3,667	3,617
2. Spent Shale (Ash)g.	2,326	2,396	2,357	2,465
3. Products (Total)g.	<u>1,265</u>	<u>1,236</u>	<u>1,310</u>	<u>1,152</u>
a. Oilg.	325.8	319.3	358.0	303.0
b. Water in Sampleg.	52.5	53.5	53.6	52.9
c. Water in Spent Shaleg.	(4.4)	(4.4)	(4.4)	(4.4)
d. Water in Air Feedg.	2.0	1.9	1.9	3.0
e. Total Water in Condenserg.	250.0	255.0	295.0	360.0(a)
f. Net Water of Combustiong.	199.9	204.0	244.1	314.5
g. H ₂ from Shaleg.	22.2	22.7	27.2	35.0
h. Net Condensed Productsg.	396.1	393.0	429.9	386.5
i. Vent Gas and Lossg.	868.9	843.0	876.6	766.5
j. Volume Vent Gasstd cu ft	23.8	22.2	17.9	15.7
k. Calc. Mol. Wt. of Vent Gas	30.55	31.80	41.20(b)	37.10(b)

Remarks:

(a) Water volume is believed to be high because of water remaining in condensing equipment from previous run.

(b) Calculated molecular weights are believed to be high; air input rates (used in determining molecular weights) are believed to be too high by 10 to 15 percent.

Figure 8. Comparison of Laboratory Runs with Data From Ruark and others (1956), p. 15); NTU Retorting Rate vs. air and recycle gas input.

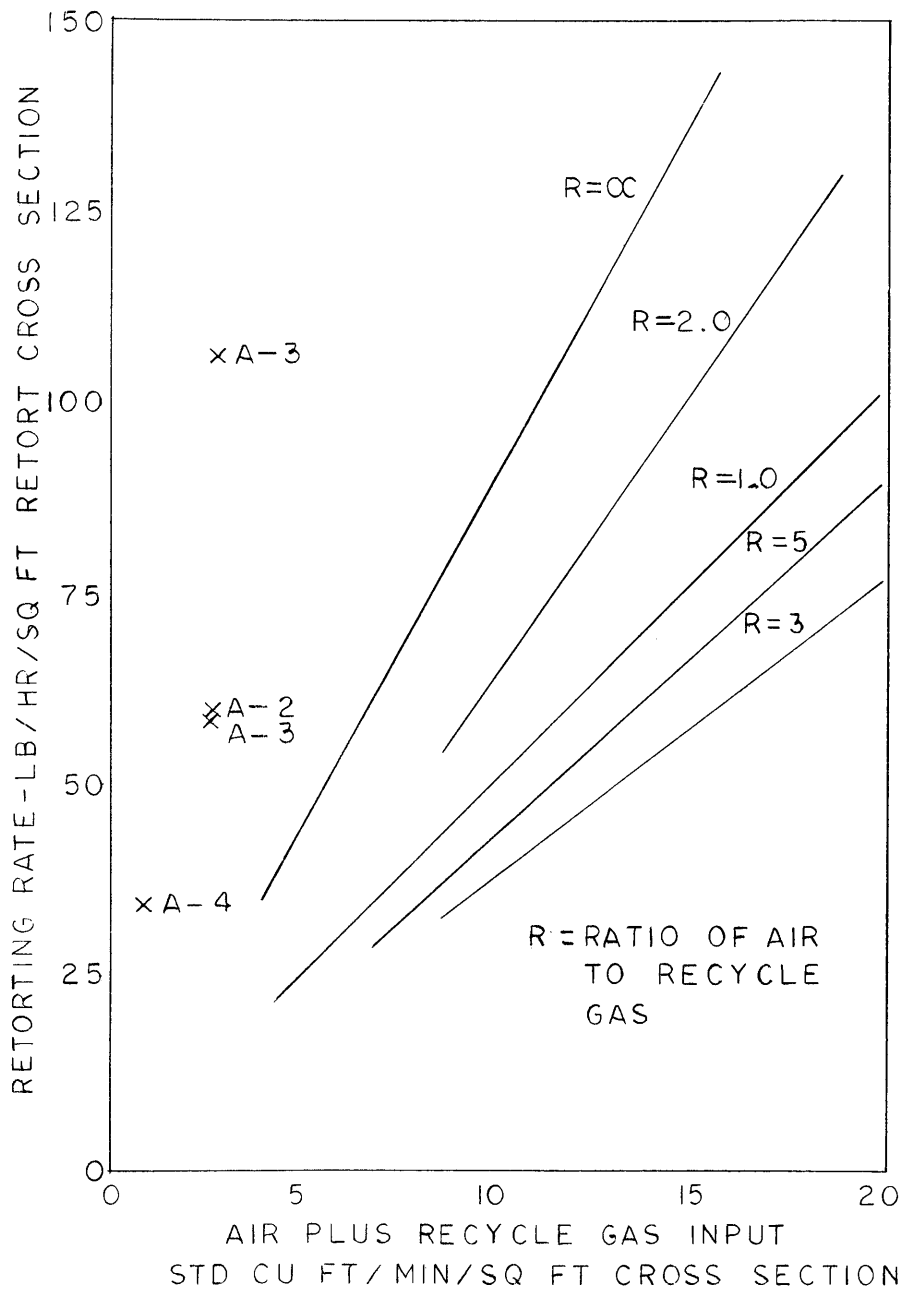
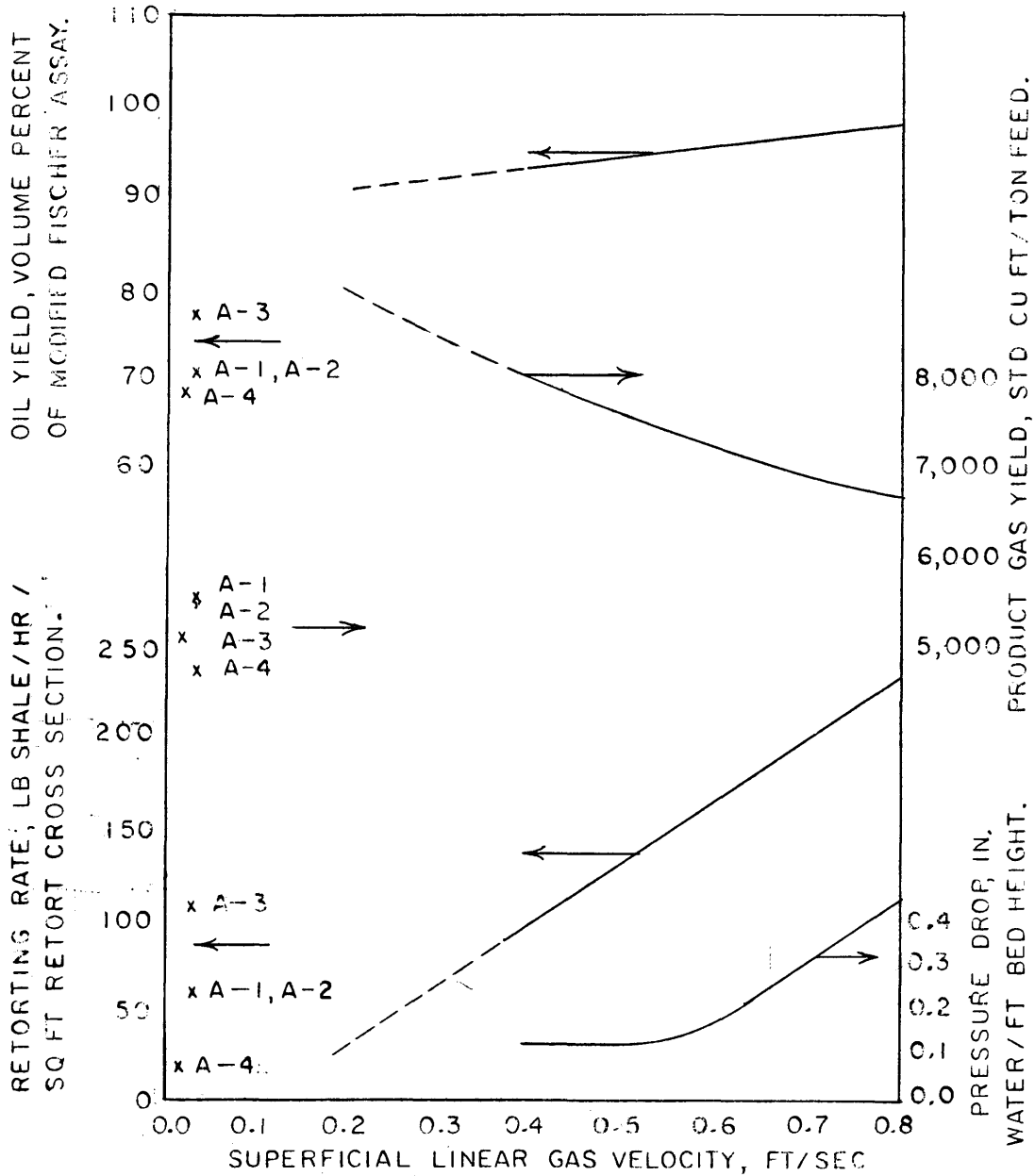


Figure 9. Comparison of Laboratory Runs With Data From U. S. Bur. Mines Rept. Inv. 4771 (1951, p. 23): Gas-Combustion retort-effect of gas velocity on retort operations.



NOTE: U.S. BUR. MINES DATA ARE FOR 20 GAL OIL / TON SHALE SIZE = + 1/2 TO - 1 IN. , GAS-AIR RATIO = 4 TO 3.

REVERSE-CURRENT BATCH RETORTING

A reverse-current run in which hot educt gas from the primary batch was passed directly to the condensing system is shown by a dashed line in Figure 6, page 37. Reverse-current retorting runs in this investigation are discussed under (1) Runs, and (2) Results.

Runs

Four reverse-current runs are reported in this investigation. Runs B#2, B#3, B#4, and B#6 were performed at air-flux rates of 48.5-, 211.6-, 168.5-, and 84.5 std cu ft/sq ft/hr. Difficulty was experienced in condensing the product oil in all the reverse-current runs. In Runs B#2, B#3, and B#4, approximately 60 percent of the condensed oil was collected as an emulsion in the primary

water trap. Oil consistently bypassed the condensing system even though ice was packed around the primary condenser -- in co-current runs, condensation took place with the primary condenser kept at 80°F. In Run B#6 ice was packed around all three condensers; in this case 95 percent of the condensed oil was collected in the primary condenser trap.

Results

The results of the four reverse-current runs are given on Table 3 and 4 on pages 53 and 54, respectively. Tables 11, 12, 13, and 14 on pages 74, 76, 78, and 80, respectively, give a time analysis with special remarks for each run.

Figure 10, page 55, shows a comparison of yields (volume percent of Fischer assay) for the laboratory runs with those of the Marathon Oil Company reverse-current experiments at the same air-flux (std cu ft/sq ft/hr) and superficial gas velocities (ft/hr). It is shown in this comparison that much poorer yields (16.0 as compared with 60.0+ percent of Fischer assay) are obtained in this

investigation than extrapolated data from Marathon Oil Company charts would infer. This outcome is probably due to the much lower pressures used in these laboratory runs (less than two atmospheres) than in the Marathon Oil Company experiment. Another contributing factor might be the difference in the shale sizes investigated: these laboratory runs used 1/2-in. minus raw feed-shale whereas Marathon Oil Company used -4 to + 8 mesh raw feed-shale.

Figure 11, page 56, illustrates a comparison of oil gravities ($^{\circ}$ API) for oil from this investigation with oil from the Marathon Oil Company reverse-combustion experiments at the same air-flux (std cu ft/sq ft/hr). It is shown in this comparison that much poorer oil (15.0 compared with 35+ $^{\circ}$ API) is obtained in this investigation that extrapolated data would infer.

Very high volumes of water (85.3 to 87.9 percent of the total condensed products) were obtained in this investigation. This fact indicates that under the conditions of this investigation large percentages of the organic matter in the oil shale are burned during

retorting to form water. In run B#6, it was noted that the product water was strong in ammonia. Tests showed a 1.4 normal basic solution.

TABLE 3
REVERSE-CURRENT SUMMARY

<u>ITEM</u>	<u>RUN NUMBER</u>	<u>B#2</u>	<u>B#3</u>	<u>B#4</u>	<u>B#6</u>
1. Fischer Assay, Feed.....	gal/ton	33.3	33.3	33.3	33.3
2. Assay of Spent Shale.....	gal/ton	0.00	0.00	0.00	0.00
3. Percent Spent to Raw Shale.....		74.5	67.8	66.7	67.7
4. Oil Recovered.....	gal/ton	4.4	5.3	5.1	4.3
5. Oil Rec. Percent Fischer Assay.....		13.1	16.0	15.2	12.9
6. Oil Gravity.....	°API @ 60°F	10.0	13.5	8.0	12.0
7. Oil Pour Point.....	°F	65	65	65	65
8. Water Condensed.....	gal/ton	31.9	30.6	31.2	30.8
9. Percent Water in Condensed Products.....		87.9	85.3	86.0	87.8
10. Maximum Retort Temperature, °F.....		1,400	1,202	1,285	1,253
11. Air Rate.....	std cu ft/min	0.063	0.275	0.219	0.110
12. Superficial Gas Velocity.....	ft/hr	53.0	202.6	143.8	78.5
13. Air Flux.....	std cu ft/sq ft/hr	48.5	211.6	168.5	84.5
14. Air Flux.....	std cu ft/ton	5,045	16,320	11,100	8,950
15. Gas Yield.....	std cu ft/ton	5,050	6,740	7,040	6,280
16. Firing Time.....	hr	5.27	3.92	3.30	3.75
17. Length of Run.....	hr	6.28	5.00	4.67	4.67
18. Rate of Retorting.....	ft/hr	0.284	0.382	0.455	0.321
19. Rate of Retorting.....	lb/hr/sq ft	19.1	25.9	30.4	27.3

TABLE 4

REVERSE-CURRENT MATERIAL BALANCE

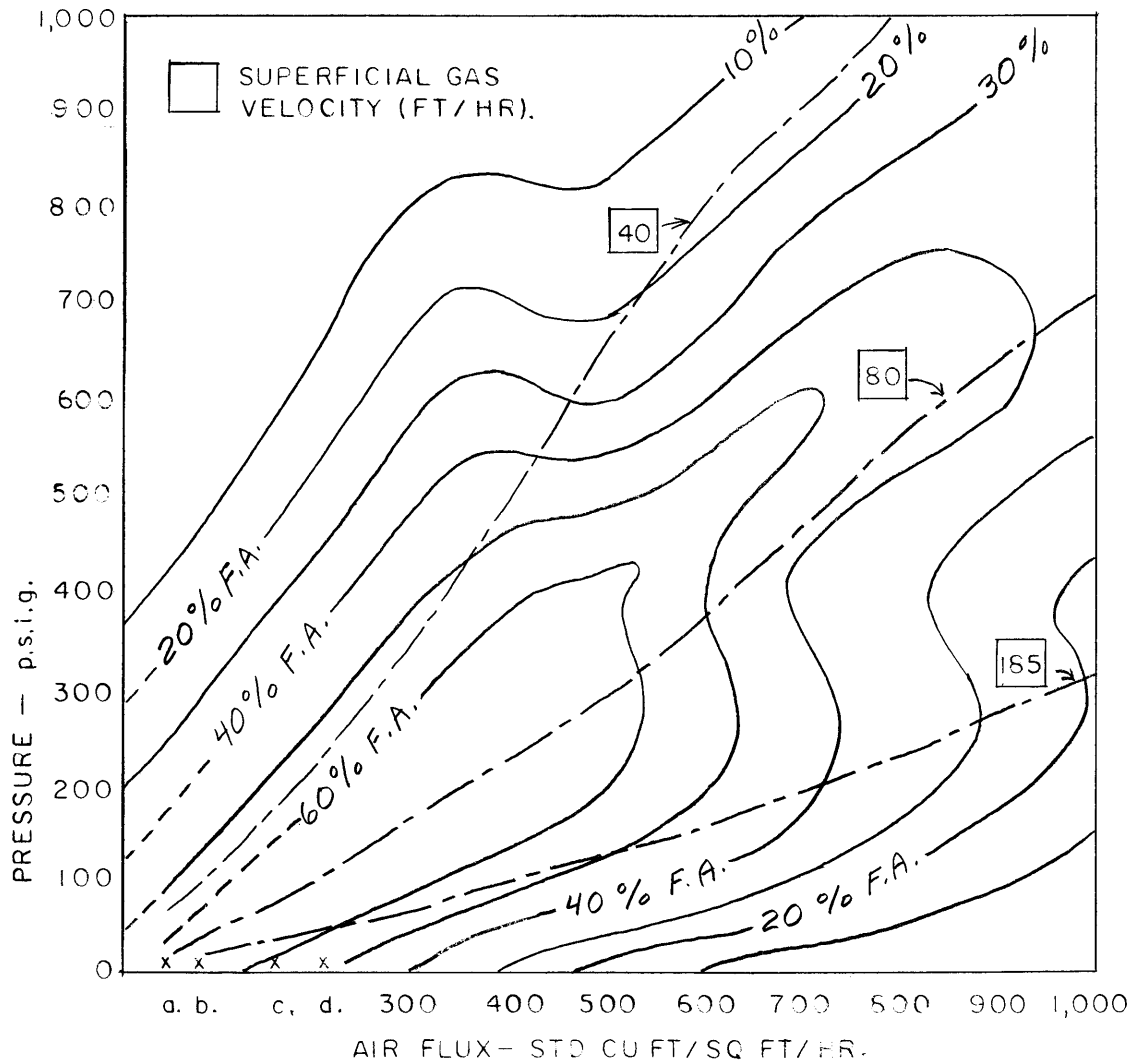
ITEM	RUN NUMBER	B#2	B#3	B#4	B#6
1. Raw Feed Shale.....g.		3,562	3,590	3,546	3,618
2. Spent Shale (Ash).....g.		2,658	2,432	2,361	2,444
3. Products (Total).....g.	904(a)	<u>1,158</u>		<u>1,185</u>	<u>1,174</u>
a. Oil.....g.		65.0	78.1	75.0	65.0
b. Water in Sample.....g.		52.1	52.5	52.1	52.7
c. Water in Spent Shale.....g.		(4.2)	(4.2)	(4.2)	(4.2)
d. Water in Air Feed.....g.		1.0	7.5	8.0	1.0
e. Est. Water from Kerogen.....		37.2	32.1	32.1	37.9
f. Total Water in Condenser.....		475.0	460.0	462.0	465.0(b)
g. Net Water of Combustion.....		389.1	372.1	374.0	377.6
h. H ₂ from Shale.....g.		42.3	41.4	41.5	41.8
i. Net Condensed Products.....g.		193.3	199.9	196.9	193.2
j. Vent Gas and Loss.....g.		710.7	959.1	988.1	980.8
k. Volume Vent Gas.....std cu ft		22.6	24.0	27.5	25.0
l. Calc. Mol. Wt. of Vent Gas.....		30.10	30.10	30.07	32.80

Remarks:

(a) Carbonates are probably not decomposed to the extent they are in the other three runs.

(b) A water analysis showed 1.4 Normal NH₄⁺.

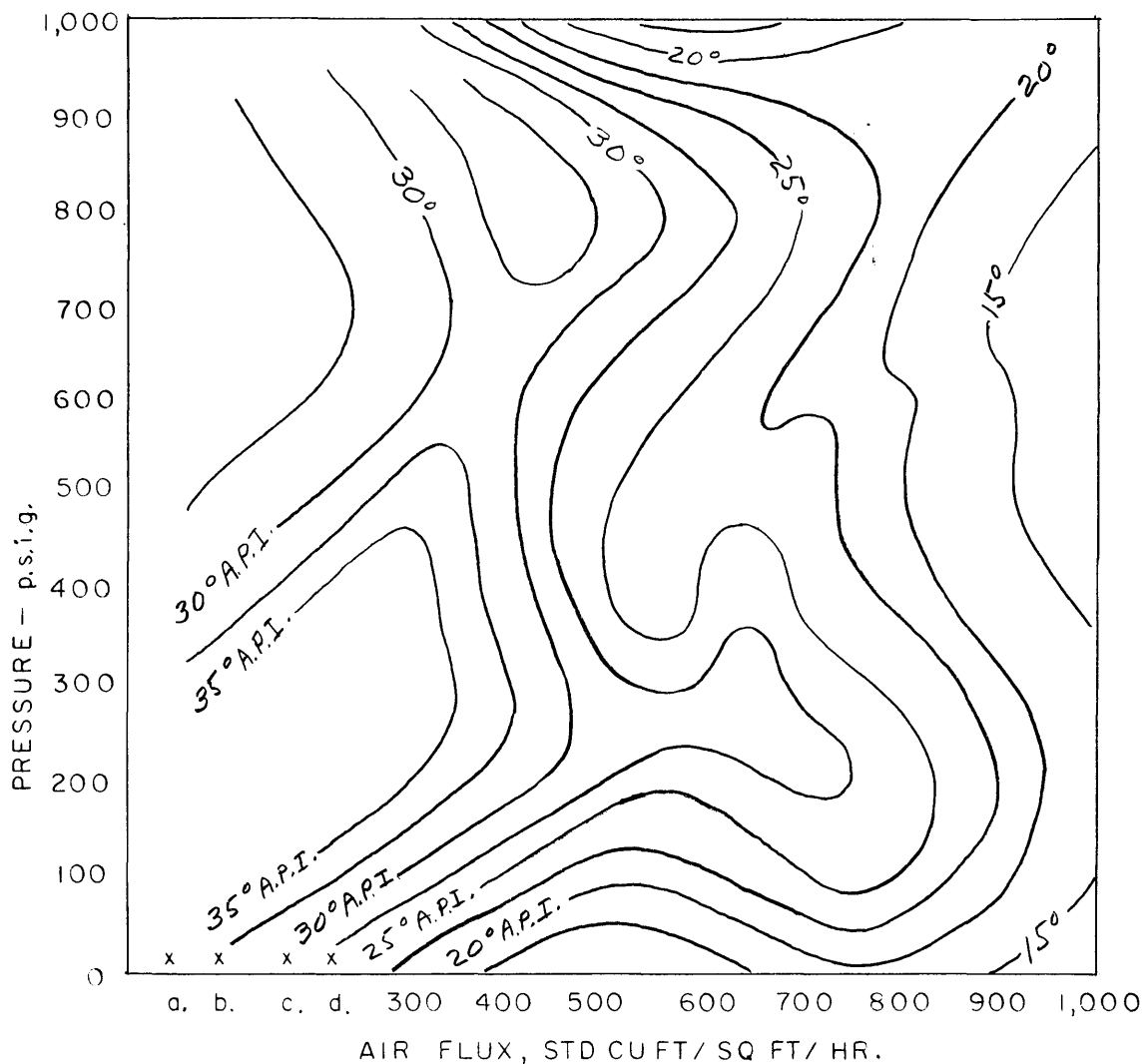
Figure 10. Comparison of Laboratory Runs with Data from Allred and Nielson (1965, p. 65): Shale Oil Yield Contours (Percent of Fischer Assay) For -4 to + 8 Mesh Particles.



DIFFERENCES IN READINGS:

- a. RUN B-2, 13.1 INSTEAD OF 60+ PERCENT OF FISCHER ASSAY.
- b. RUN B-6, 12.9 do. do. 60+ do. do. do. do.
- c. RUN B-4, 15.2 do. do. 60+ do. do. do. do.
- d. RUN B-3, 16.0 do. do. 50+ do. do. do. do.

Figure 11. Comparison of Laboratory Runs
 With Data From Allred and Nielson (1965, p. 66):
 Shale Oil Gravity Contours (°API)



DIFFERENCES IN READINGS:

- a. RUN B-2, 10.0 INSTEAD OF 32+ DEGREES API GRAVITY.
- b. RUN B-6, 12.0 do. do. 32+ do. do. do.
- c. RUN B-4, 8.0 do. do. 28+ do. 10. do.
- d. RUN B-3, 13.5 do. do. 25+ do. do. do.

ANALYSIS OF RAW FEED-SHALE AND SPENT SHALE

The raw feed-shale used in this investigation came from the Mahogany marker zone of the Green River Formation near Rifle, Colorado. It was obtained from the Colorado School of Mines Research Foundation at Golden, Colorado. The shale contained 33.3 gal oil per ton and 3.50 gal water per ton as determined by a Fischer assay performed by the Colorado School of Mines Research Foundation on March 30, 1966.

A sieve analysis of the raw feed-shale as obtained is given in Table 5, page 60, and a grain size distribution curve is shown on Figure 12, page 59. The sieve analysis shows that the raw feed-shale is crushed to 1/2-in. minus with the majority of the rock crushed to sizes between 0.500 and 0.185 in. The raw feed-shale contains

approximately 2.7 percent fines.

Fischer assays made by the Colorado School of Mines Research Foundation on March 30, 1966 of the spent shale from the present investigation show 0.00 gal oil per ton (all carbon burned) and 0.29 gal water per ton for representative samples of co-current spent shale, and 0.00 gal oil per ton (all carbon burned) and 0.28 gal water per ton for representative samples of reverse-current spent shale. The spent shale from both co-current and reverse-current burning is a very clean, white ash with no clinkering. Weight-loss calculations (not confirmed by assay) indicate that most of the mineral carbonates in the raw feed-shale are decomposed during both co-current and reverse-current burning runs. The spent shale for both averaged approximately 65 percent by weight of the raw feed shale.

Figure 12. Raw Shale Feed Sieve Analysis

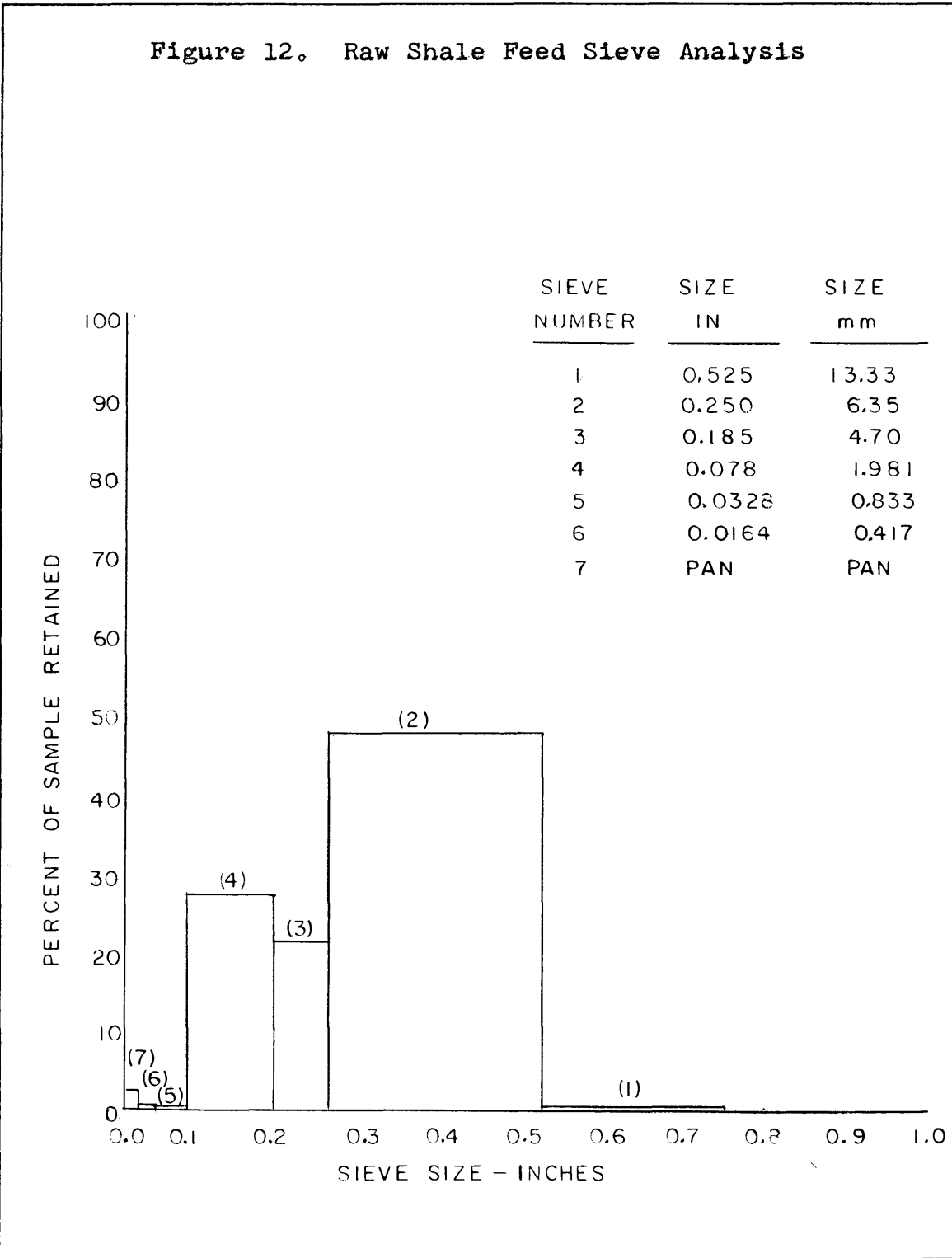


TABLE 5

RAW SHALE-FEED SIEVE ANALYSIS

33.3 GAL/TON COLO. SHALE

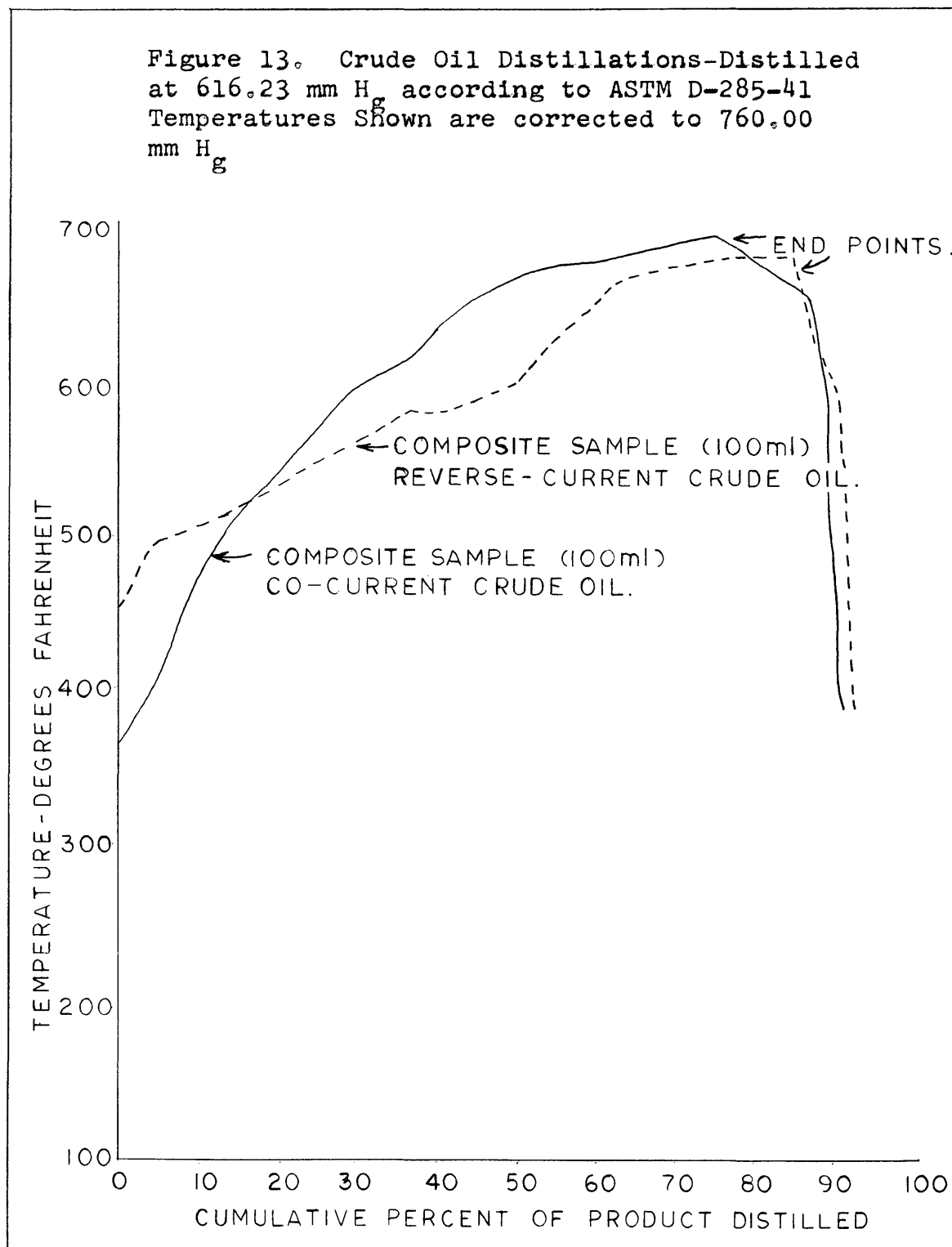
<u>Sieve Number</u>	<u>Sieve Size-mm</u>	<u>Weight Sieve-g</u>	<u>Weight Sieve and Sample-g</u>	<u>Weight Sample-g</u>	<u>Percent Retained</u>
1	13.33	624.9	630.0	5.1	0.25
2	6.35	734.9	1,699.5	964.6	47.55
3	4.70	600.8	1,037.4	436.6	21.50
4	1.981	497.9	1,049.2	551.3	27.15
5	0.833	485.6	984.7	9.1	0.45
6	0.417	467.1	476.8	9.7	0.47
Pan	---	<u>381.1</u>	<u>436.5</u>	<u>55.4</u>	<u>2.73</u>
Total		3,792.3	5,842.1	2,031.8	100.00

Date of Test - April 5, 1966
Place - CSM Soils Laboratory

DISTILLATION OF CRUDE OIL SAMPLES

Two 100-ml composite samples (one co-current and one reverse-current) were distilled according to A.S.T.M. Designation D 285-41. Summaries of the distillations are given on Table 6, page 63, and distillation curves are shown on Figure 13, page 62.

It is shown on the distillation curves that the reverse-current composite crude-oil sample has fewer low-boiling-point ends, more intermediate-boiling-point ends, and about the same number of high-boiling-point ends as the co-current composite crude-oil sample has.



Time	Co-Current Sample			Reverse-Current Sample		
	Temp. °F	Volume Distillate cc	Bath Temp. °F	Temp. °F	Volume Distillate cc	Bath Temp. °F
5:25	100	0	72	146	0	72
5:35	115	0	72	167	0	72
5:43	130	0	72	360	0	72
5:46				490	(1st Drop)	72
5:50				480	4	72
5:54	300	0	72	485	7	72
5:56	360	(1st Drop)	72	475	11	72
5:59	380	3	72	495	13	72
6:01	400	5	72	515	19	72
6:03	416	6	72	524	22	72
6:06	457	9	72	541	29	72
6:08	488	13	72	554	33	72
6:10	515	18	72	560	36	72
6:12	544	23	72	562	40	72
6:14	568	28	72	568	43	72
6:16	584	32	75(a)	570	46	92(a)
6:18	596	36	80	568	49	92
6:20	618	40	88	582	50	92
6:22	622	42	94	604	53	92
6:24	637	46	102	625	58	92
6:26	648	50	106	642	62	92
6:28	655	55	110	654	68	97
6:30	660	61	117	660	75	108
6:33	668	68	117	660	84	108
6:35	672	74	117	570(b)	90	108
6:37	655(c)	80	117			
6:39	640	84	117	318(d)	93	107

TABLE 6 -- CRUDE OIL DISTILLATIONS (Continued)

Time	Co-Current Sample		Reverse-Current Sample	
	Temp. °F	Volume cc	Temp. °F	Volume cc
6:40	638	86	117	
6:43	582	88		
6:45	410(e)	90		

Remarks :

- (a) Hot water added to condenser baths.
- (b) Heavy white smoke forming.
- (c) Heavy yellow to deep-red smoke forming.
- (d) Heavy wax and carbon remaining in distillation flask.
- (e) Heavy wax and carbon remaining in distillation flask.

CONCLUSIONS

1. The batch retort used in this investigation is satisfactory for comparing co-current with reverse-current retorting in laboratory experiments.

2. At the pressures and air-flux rates used in this investigation, co-current retorting yields:

- a. A dark-green oil.
- b. A medium-weight oil (22.7°API).
- c. An intermediate-pour-point oil (75 °F).
- d. High percentages of ammonia water (40 percent by volume of the total condensed products; a 0.4 normal ammonia solution).
- e. Low to intermediate mass rates (58.7 to 106.3 lb/hr/sq ft retort cross section).
- f. Intermediate yields (70 percent of Fischer assay oil recovery).
- g. Advanced decomposition of mineral carbonates (the spent shale is 65 percent by weight of the raw feed-shale).

3. At the pressures and air-flux rates used in this investigation, reverse-current retorting yields:

- a. A black oil.
- b. A high-weight oil (12.0 °API).
- c. An intermediate-pour-point oil 65 °F).
- d. Very high percentages of ammonia water (85 percent by volume of the total condensed products; a 1.4 normal ammonia solution).
- e. Low mass rates (19.4 to 30.4 lb/hr/sq ft retort cross section).
- f. Very low yields (12.9 to 16.0 percent of Fischer assay oil recovery).
- g. Advanced decomposition of mineral carbonates (the spent shale varied between 66.7 and 74.5 percent by weight of the raw feed-shale).

4. Crude oil distillations of composite samples show that reverse-current oil obtained in this investigation has fewer low-boiling-point ends, more intermediate-boiling-point ends, and about the same number of high-boiling-point ends as the co-current oil has.

5. Reverse-current retorting does not appear to be an attractive method for surface retorting where low pressures necessarily encountered would probably give the same results as those found in this investigation.

6. By using air-flux rates that are about half those reported being used in other co-current retorting methods, it is possible to obtain larger volumes of ammonia water and a better quality oil (higher API gravity and lower pour point) than are obtained in the other methods. But, when air-flux rates are lowered there is a loss of oil yield (percent of Fischer assay) and a loss of mass rate (lb/hr/sq ft retort cross section).

APPENDIX

Tables 7 through 14.

TABLE 7

RUN A#1 (CO-CURRENT)

RUN A#1, MARCH 17, 1966
CO-CURRENT BURNING

ROBERT E. CRUMB
MASTER OF SCIENCE THESIS

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
11:14		460	82	Start furnace.
11:53				Drain product from condenser trap; obtain 100 ml water and oil. Shut off fuel; ignite batch.
11:58		500	"	
12:05	163.0(a)	785	"	
12:08	"	880	"	
12:14	"	950	"	Smoke to bottles.
12:25	"	885	81	
12:36	"	860	80	Raise temperature probe 5 1/2" for following reading.
12:53	"	980	"	
1:10	"	882	"	
1:45	"	770	76	Shut down. Heat condensers to 128°F. for 20 min to speed collection of free oil from condenser trap.

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.212 std cu ft/min.
 2. 163.0 std cu ft/sq ft retort cross section/hr.
 3. 5,570 std cu ft/ton shale retorted.

TABLE 8

RUN A#2 (CO-CURRENT)

ROBERT E. CRUMB
MASTER OF SCIENCE THESIS

RUN A#2, MARCH 17, 1966
CO-CURRENT BURNING

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
3:00				
3:59		492	80	Start furnace.
4:02	163.0(a)	575	80	Shut off fuel; ignite batch.
4:07	"	798	84	
4:19	"	985	81	
4:24	"	982	82	
4:27	"	960	"	
4:30	"	1012	78	Temperature probe raised 5 1/2" for following readings.
4:40	"	1049	82	Smoke to bottle traps.
4:50	"	1062	78	
4:55	"	1062	"	Maximum temperature recorded during run.
5:11	"	1010	82	
5:28	"	925	80	
5:45	"	860	81	Shut down. Heat condensers to 140°F to help speed collection of free oil in condenser trap.

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.212 std cu ft/min.
 2. 163.0 std cu ft/sq ft retort cross section/hr.
 3. 5,450 std cu ft/ton shale retorted.

TABLE 9

RUN A#3 (CO-CURRENT)ROBERT E. CRUMB
MASTER OF SCIENCE THESISRUN A#3, MARCH 17, 1966
CO-CURRENT BURNING

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
6:24		382	83	Start furnace.
6:50		500	82	
7:00		600	74	Shut off fuel; ignite batch.
7:05	163.0(a)	940	80	
7:18	"	962	"	Smoke to bottle traps.
7:23	"	932	81	Temperature probe raised 5 3/4" for following readings.
7:32	"			Maximum temperature recorded during run.
7:40	"	1132	81	
7:50	"	1118	"	
8:00	"	1042	"	
8:15	"	921	"	
8:25	"	880	"	
8:30	"	860	"	Shut down. Condensers heated to 140°F for 1/2 hr. to help speed collection of free oil in condenser trap.

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.212 std cu ft/min.
 2. 163.0 std cu ft/sq ft retort cross section/hr.
 3. 4,770 std cu ft/ton shale retorted.

TABLE 10

RUN A#4 (CO-CURRENT)

ROBERT E. CRUMB MASTER OF SCIENCE THESIS	RUN A#4, APRIL 2, 1966 CO-CURRENT BURNING			
<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP °F</u>	<u>REMARKS</u>
7:25		160	64	Start furnace; run hot gas to dehydrator.
8:05		455	"	Run hot gas to batch.
8:30		525	"	
8:35	82.0(a)			Shut off fuel; ignite batch. Temperature probe raised 12" to establish temperature profile. Drain product from condenser trap; obtain 25 ml water, no oil.
8:45	"	270	"	Temperature reading at top of batch is 215°F.
9:00	"	220	65	
9:15	"	238	64	
9:32	"	322	"	
9:45	"	420	"	
10:00	"	574	"	
10:36	"	942	"	
10:46	"	990	"	
11:00	"	1020	"	Maximum temperature reading recorded during run. Temperature reading at bottom of batch is 664°F.
11:15	"	1005	"	

TABLE 10 (Continued)

RUN A#4 (CO-CURRENT)

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER</u>		<u>REMARKS</u>
			<u>TEMP. °F</u>	<u>TEMP. °F</u>	
11:30	82.0(a)	954	64		Shut down. Condensers heated to 140°F for 1/2 hr to help speed collection of free oil in condenser trap.
11:35	"	920	"		

ROBERT E. CRUMB
MASTER OF SCIENCE THESIS

RUN A#4, APRIL 2, 1966
CO-CURRENT BURNING

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.144 std cu ft/min.
 2. 64.1 std cu ft/sq ft retort cross section/hr.
 3. 5,100 std cu ft/ton shale retorted.

TABLE 11

RUN B#2 (REVERSE-CURRENT)

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
10:18				Start furnace; run hot gas to dehydrator.
10:45		110	80	Run hot gas to baten.
11:22		435	82	Drain product from trap; recover 30 ml water.
11:31		476	74	
11:39		500	"	Shut off fuel; ignite batch.
11:43	48.5(a)	610	78	Drain product from trap; recover 80 ml water.
11:49	"	782	74	Reverse air direction; run by reverse current.
11:53	"	840	72	Smoke to water traps.
12:04	"	958	65	
12:10	"	930	63	
12:15	"	927	62	
12:59	"	785	"	Raise temperature probe 12" for remaining readings.
1:04	"	442	"	
1:20	"	438	64	
1:40	"	550	"	
2:10	"	758	"	
2:32	"	842	62	

ROBERT E. CRUMB
 MASTER OF SCIENCE THESIS
 RUN B#2, MARCH 21, 1966
 REVERSE-CURRENT BURNING

TABLE 11 (Continued)

RUN B#2 (REVERSE-CURRENT)

ROBERT E. CRUMB
 MASTER OF SCIENCE THESIS
 RUN B#2, MARCH 21, 1966
 REVERSE-CURRENT BURNING

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
3:09	48.5(a)	868	62	
3:35	"	1022	63	
3:43	"	1400	"	Maximum temperature observed.
4:20	"	1010	62	
5:05	"	868	"	Shut down run; no additional oil being recovered.

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.063 std cu ft/min.
 2. 48.5 std cu ft/sq ft retort cross section/hr.
 3. 5,045 std cu ft/ton shale retorted.

TABLE 12

RUN B#3 (REVERSE-CURRENT)

TIME	AIR FLUX	BATCH TEMP. °F	CONDENSER TEMP. °F	REMARKS
7:15		265	75	Start furnace run hot air to batch.
8:02				Drain product from condenser recover 100 ml water.
8:08		350	65	
8:14	211.6(a)	500	72	Shut off fuel gas; ignite batch.
8:20	"	700	53	Reverse air direction to batch for reverse current burning.
8:30	"	792	50	Smoke to trap bottles.
8:40	"	925	"	
9:02	"	975	47	
9:12	"	935	48	
9:30	"	382	49	Fire front apparently past probe; probe raised 12".
9:45	"	489	47	
10:00	"	645	43	
10:15	"	850	45	
10:30	"	970	43	
10:47	"	998	44	
11:02	"	1014	"	
11:15	"	1160	45	

ROBERT E. CRUMB
MASTER OF SCIENCE THESIS

RUN B#3, MARCH 23, 1966
REVERSE-CURRENT BURNING

TABLE 12 (Continued)

RUN B#3 (REVERSE-CURRENT)

ROBERT E. CRUMB
 MASTER OF SCIENCE THESIS
 RUN B#3, MARCH 23, 1966
 REVERSE-CURRENT BURNING

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
11:31	211.6(a)	1202	45	Maximum temperature recorded during run.
11:45	"	1125	"	
12:07	"	1019	"	
12:15	"	920	"	

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.2275 std cu ft/min.
 2. 211.6 std cu ft/sq ft retort cross section/hr.
 3. 16,320 std cu ft/ton shale retorted.

TABLE 13

RUN B#4 (REVERSE-CURRENT)

TIME	AIR FLUX	BATCH TEMP. °F	CONDENSER TEMP. °F	REMARKS
5:10				Start furnace.
5:31		200		
6:02		500	34	Shut off fuel gas; ignite batch.
6:12	168.5(a)	600	40	Reverse air direction.
6:17	"			Drain condenser trap; recover 20 ml water, skim oil.
6:30	"	998	32	1st water trap is black with oil residue, 2nd water trap is milky.
6:45	"	1018	"	
7:00	"	1000	"	Fire front apparently past probe; probe raised 12".
7:15	"	380	"	
7:30	"	660	"	
7:45	"	1000	"	
8:02	"	1100	"	
8:15	"	1112	"	
8:33	"	1285	"	Maximum temperature recorded during run.
8:45	"	1255	"	1st and 2nd water traps are turbid green with approximately 100 ml oil emulsion circulation

ROBERT E. CRUMB
MASTER OF SCIENCE THESIS

RUN B#4, MARCH 24, 1966
REVERSE-CURRENT BURNING

TABLE 13 (Continued)

RUN B#4 (REVERSE-CURRENT)

ROBERT E. CRUMB
 MASTER OF SCIENCE THESIS
 RUN B#4, MARCH 24, 1966
 REVERSE-CURRENT BURNING

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
9:00	168.5(a)	1165	32	
9:15	"	1039	34	
9:30	"	910	33	Shut down.

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.219 std cu ft/min.
 2. 168.5 std cu ft/sq ft retort cross section/hr.
 3. 11,100 std cu ft/ton shale retorted.

TABLE 14

RUN B#6 (REVERSE-CURRENT)

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
4:20				
5:08	84.5(a)	500	32	Start heating batch.
5:11	"	550	"	Shut off pre-heating furnace.
5:15	"	700	"	Trap bottles smokey.
5:20	"	860	"	Air direction reversed. Batch now on reversed current burning.
5:30	"	948	"	
5:40	"	180	"	Last temperature reading at the bottom of the batch.
5:45	"	188	"	Temperature reading at a point 12" up from the bottom of the batch.
6:00	"	225	"	
6:10	"	270	"	
6:32	"	415	"	
6:45	"	540	"	Oil visible in water traps, Est. 2 ml in 1st trap Est. 0 ml in 2nd trap.
7:00	"	768	"	
7:15	"	952	"	
7:40	"	1000	"	
7:47	"	1007	"	
7:55	"	1050	"	

ROBERT E. CRUMB
MASTER OF SCIENCE THESIS

RUN B#6, APRIL 2, 1966
REVERSE-CURRENT BURNING

TABLE 14 (Continued)

RUN B#6 (REVERSE-CURRENT)

ROBERT E. CRUMB
 MASTER OF SCIENCE THESIS
 RUN B#6, APRIL 2, 1966
 REVERSE-CURRENT BURNING

<u>TIME</u>	<u>AIR FLUX</u>	<u>BATCH TEMP. °F</u>	<u>CONDENSER TEMP. °F</u>	<u>REMARKS</u>
8:02	84.5(a)	1153	32	
8:15	"	1253	"	
8:30	"	1125	"	
8:45	"	1010	"	
9:00	"	891	"	Fire front past probe; batch shut down to cool.

NOTE:

- a. Air rates were continuously adjusted to remain constant. Rates were:
1. 0.110 std cu ft/min.
 2. 84.5 std cu ft/sq ft retort cross section/hr.
 3. 8,950 std cu ft/ton shale retorted.

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