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SOME RESULTS FROM THE OPERATION OF A 150-TON OIL SHALE RETORT

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SOME RESULTS FROM THE OPERATION OF A 150-TON OIL SHALE RETORT

by

A. E. Harak,[1] L. Dockter,[1] and H. C. Carpenter[2]

ABSTRACT

To determine the retorting characteristics of a column of mine-run, ungraded oil shale ranging in size from fines to pieces weighing as much as 5 tons, a 150-ton batch retort was designed and constructed at the Laramie Energy Research Center, Laramie, Wyo. The retort vessel is 45 feet high by 11-1/2 feet in inside diameter and is supported in a 92-foot-high steel superstructure. Mine-run shale from the Bureau of Mines facility near Rifle, Colo., assaying about 25 gallons of oil per ton, is currently being used as the test shale. To date, three runs have been completed. Results of the second and third runs are reported in this paper.

The two runs considered in this report are the first of a series of nine to determine operating conditions that will yield maximum oil recovery from a column of mine-run shale. Variables studied are the oxygen content of the retorting gas and the space velocity of the retorting gas expressed as ft^3 gas/ft^2 bed/min. Oil recoveries for the two runs reported are 62.2 volume percent of Fischer assay for the run using higher oxygen and space velocity, compared with 39.3 volume-percent when less oxygen and a lower space velocity were used.

INTRODUCTION

During recent years the Bureau of Mines has been actively engaged in research to develop a technology for the recovery of shale oil from a mass of shale rubble resulting from a nuclear explosion or other more conventional means. To determine the retorting characteristics of a column of shale rubble, including pieces weighing as much as 5 tons, the Laramie Energy Research Center has operated a nominally sized, 150-ton retort for the past year. While

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this retort is considerably smaller than a nuclear chimney,[3] results from this experimentation should yield valuable background information as to retorting characteristics of a shale column having a wide range of particle sizes. This information will be useful in designing future in situ processes for oil shale formations fractured by nuclear or more conventional means.

This progress report discusses the design, operating procedure, and results from the second and third runs using a 150-ton oil shale retort. Because the first run with the 150-ton unit was a shakedown run, the results are not included in this report. A paper discussing the first run was presented at the Sixth Oil Shale Symposium in Denver, Colo., in February 1970.

RETORT DESIGN

The 150-ton, batch-type retort shown in figure 1 is a refractory-lined carbon steel cylinder with an opening at the top for shale loading and a hinged grate at the bottom for supporting the shale bed. The refractory lining consists of 6-inch-thick firebrick, backed up by 4 inches of insulating castable refractory.

The retort vessel is 45 feet high by 11-1/2 feet in inside diameter and is supported in a 92-foot-high steel superstructure. The hinged grate is opened by a hydraulic mechanism to discharge spent shale from the vessel after the termination of a run.

The flow diagram of the 150-ton retort is shown in figure 2. A natural gas burner mounted on the retort lid is used to initiate combustion of the shale bed. Roots-type blowers[4] are used to force air into the retort and downward through the shale bed. An oil tank, mounted on load cells, collects liquid products from the retort outlet. Gaseous products from the retort, which contain some oil and water mist, are passed through a packed tower and a demist tank to remove most of the entrained material. After passing through a Roots-type blower, the gas stream splits: Some can be recycled back into the retort; the remainder vents to the stack. The stack, which is equipped with a natural gas burner, is used to oxidize any combustible components in the gas stream. Location of control instrumentation is also shown in figure 2.

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[3]Lekas, Mitchell A., Bruce G. Bray, Harry C. Carpenter, H. H. Aronson, Gerald U. Dinneen, and J. D. Downen. The Bronco Oil Shale Study, Part I. PNE-1400, Oct. 13, 1967, 21 pp.

Lombard, David B., Bruce G. Bray, Harold W. Sohns, Thomas S. Sterrett, Robert S. Brundage, and Harry C. Carpenter. The Bronco Oil Shale Study, Part II. PNE-1400, Oct. 13, 1967, 43 pp.

[4]Reference to specific makes or models of equipment is made to facilitate understanding and does not imply endorsement of such brands by the Bureau of Mines.

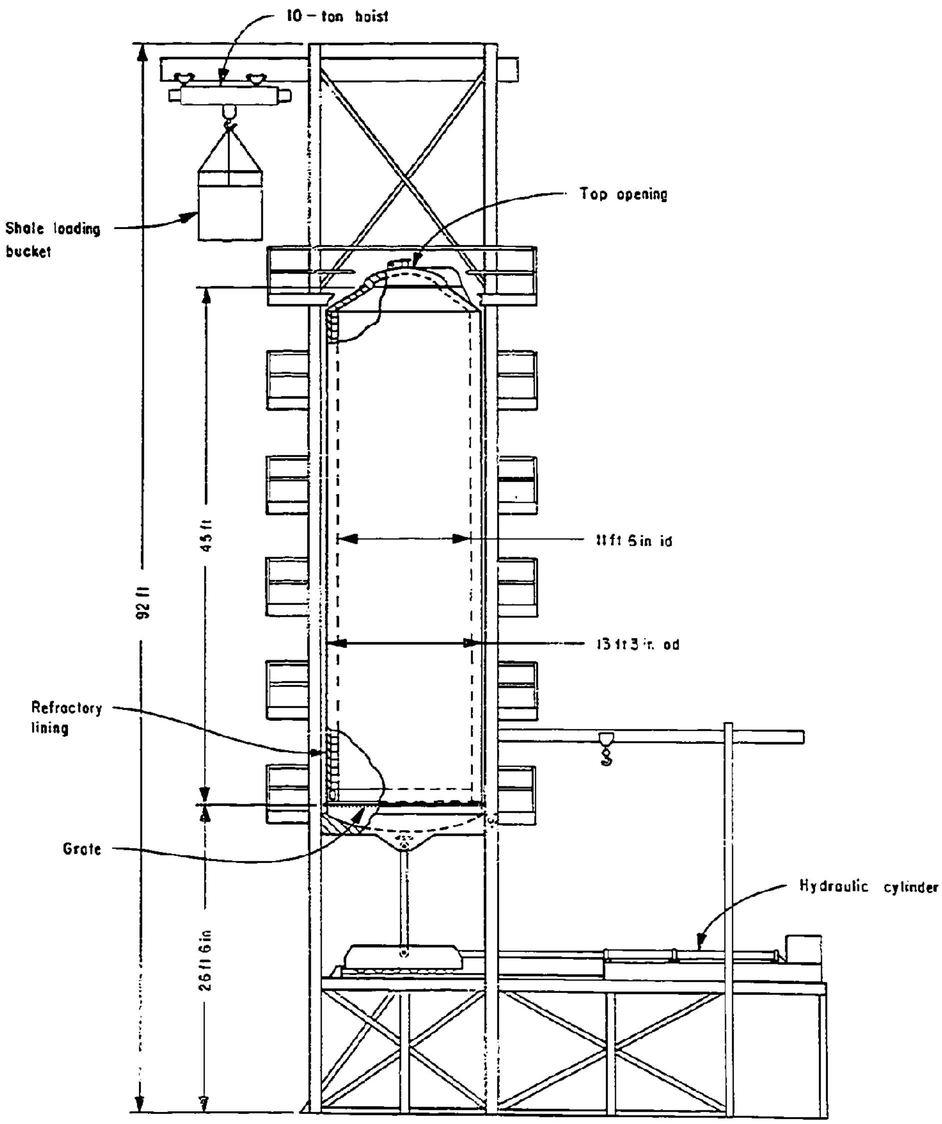

FIGURE 1. - 150-Ton Oil Shale Retort.

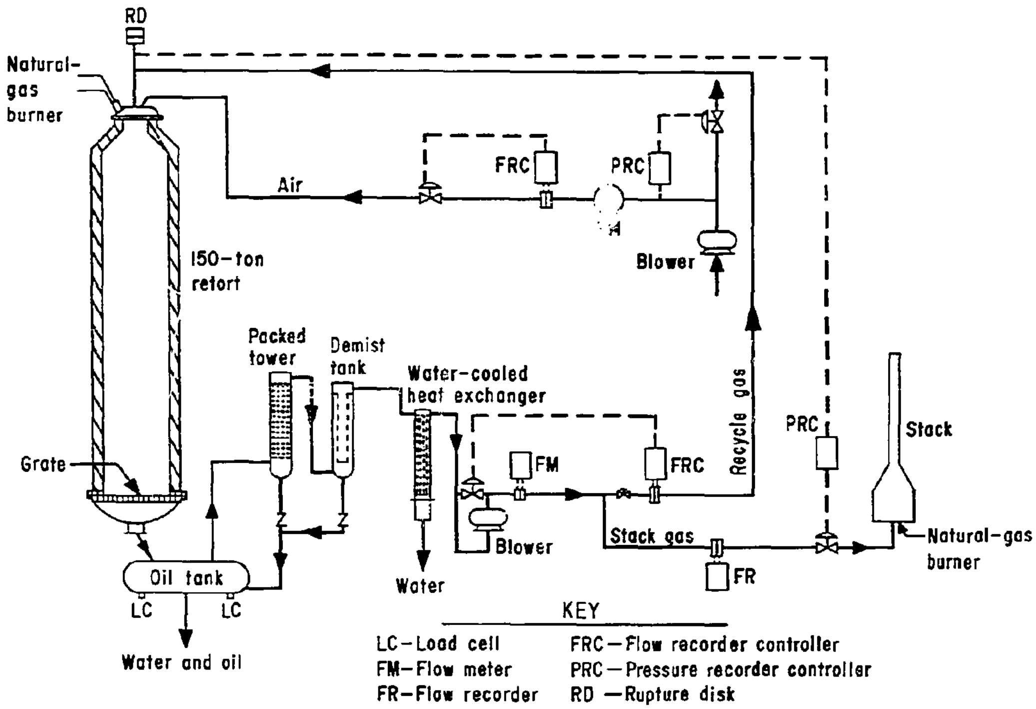

FIGURE 2. - Flow Diagram of 150-Ton Retort.

OPERATING PROCEDURE

The oil shale for these runs was obtained from a stockpiled supply at the Bureau of Mines Anvil Points facility near Rifle, Colo. A size distribution curve for the test shale is shown in figure 3. This curve was originally drawn from data obtained from screen analyses and photographic studies on the shale for run 1. Since the shale for runs 2 and 3 was obtained from the same stockpile supply, it is assumed to have approximately the same size distribution. About 20 percent of the shale had a major dimension larger than 20 inches, and about 10 percent was smaller than 1 inch. The shale for these runs was sampled at the Anvil Points facility, and the Fischer assays for runs 2 and 3 averaged 25.4 and 24.7 gal/ton, respectively.

A cylindrically shaped, 3-ton-capacity, bottom-opening bucket and a 10-ton hoist, mounted at the top of the superstructure, were used to convey shale into the retort vessel. The bucket is designed to open automatically when it bottoms, and shale is released when the bucket is raised. A 6-inch-thick layer of crushed rock, sized from 1-1/2 inches to 3 inches, was placed on the grate prior to shale loading to prevent an excessive amount of shale fines from falling through the 1-inch-wide grate openings during the loading operation. During loading, a man was periodically lowered into the retort to

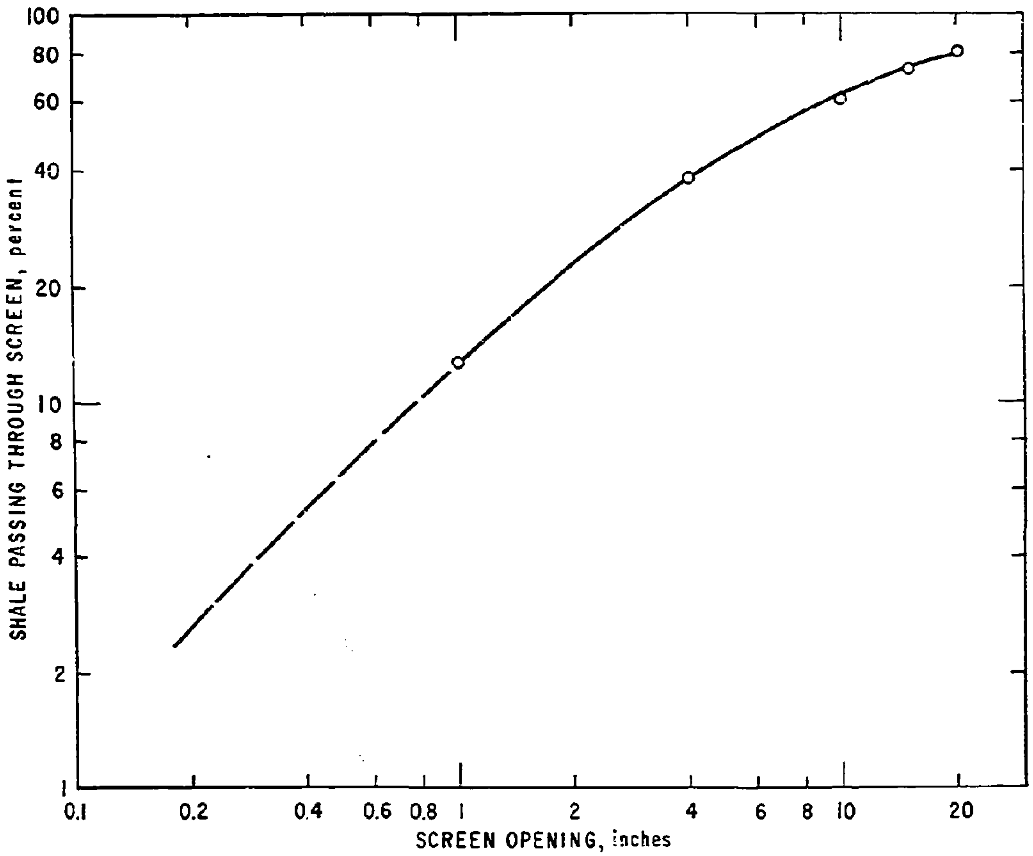

FIGURE 3. - Size Distribution of Shale Used in 150-Ton Retort.

level the bed and place thermocouples. At each 3-foot level of bed height, eight thermocouples were placed in a horizontal plane as shown in the upper right hand part of figure 4. A total of 112 thermocouples, wired to five temperature recorders, were used to log bed temperatures.

The shale charge for each run included one large block placed about 7 feet below the top of the shale bed. These blocks for runs 2 and 3 weighed 7,500 and 10,100 lb, respectively. Four thermocouples were cemented inside each block, and four were placed around each block as shown at the right side of figures 5 and 6. One was located directly on top of each block.

After the retort was loaded with shale to a bed depth of 43 feet, the vessel was closed. A natural gas burner at the top of the retort then was ignited and allowed to burn for 3 to 4 hours at a heat release rate of about 500,000 Btu/hr. After shutting off the burner, air and recycle gas were

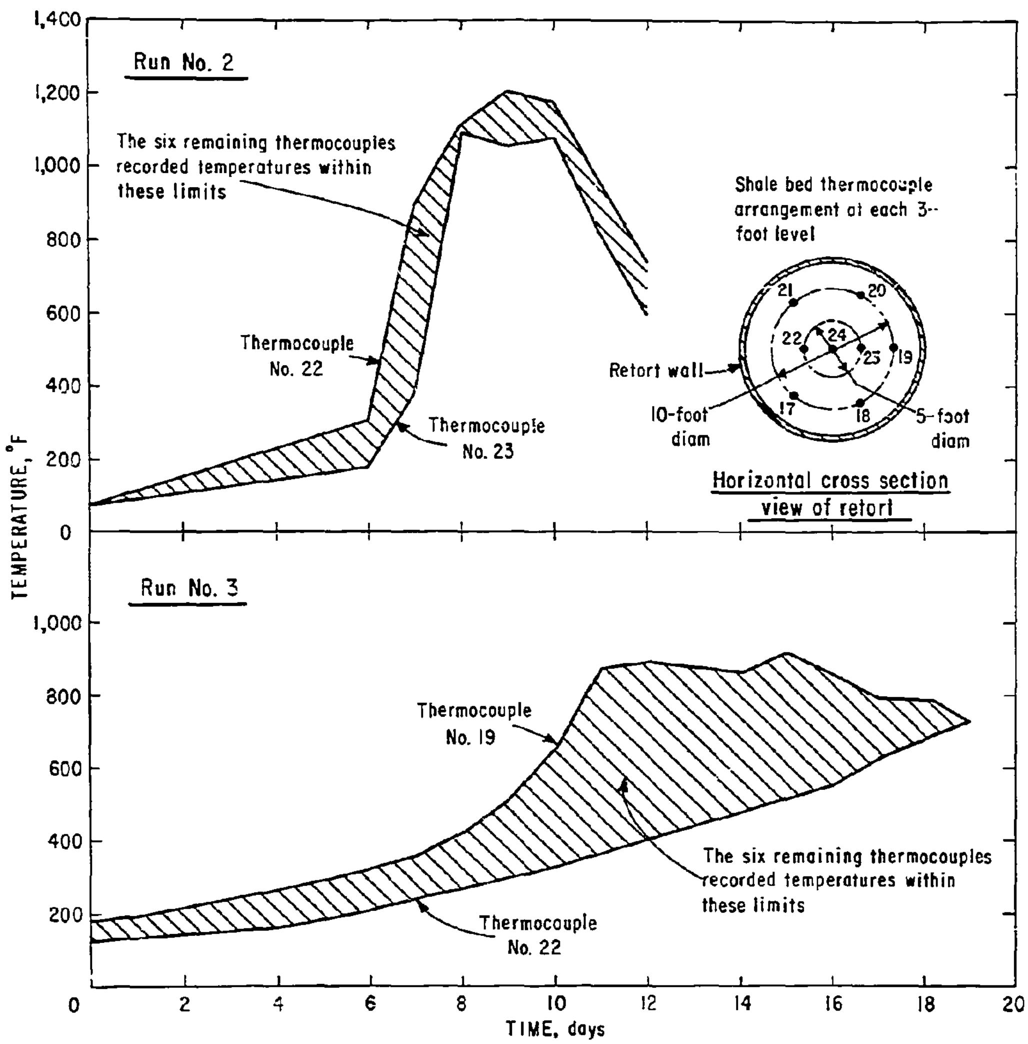

FIGURE 4. - Thermocouple Arrangement and Temperature Profile of Shale Bed 18 Feet Above the Grate for Runs 2 and 3.

started at predetermined flow rates and retorting progressed downward through the bed.

The air and recycle gas rates were held constant throughout each run. Retorting was continued until the grate temperature reached 500° F, somewhat lower than the 600° F maximum allowable design temperature. The gas flows were then shut off and the shale bed was allowed to cool.

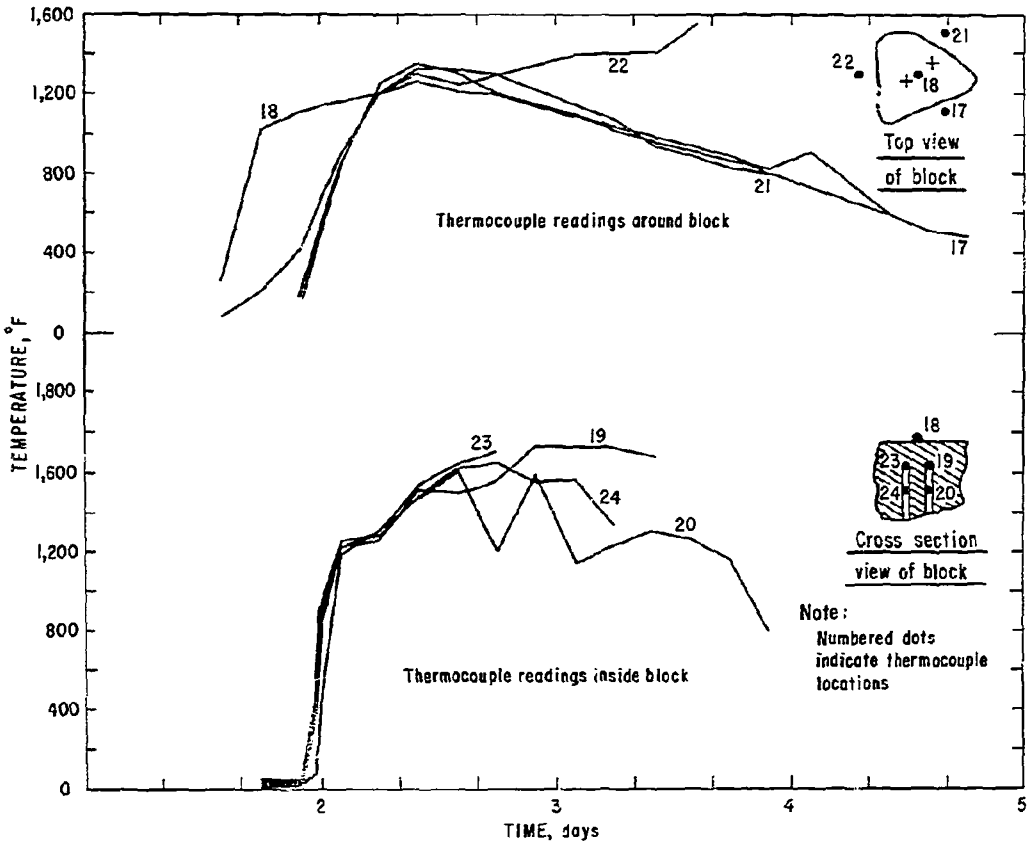

FIGURE 5. - Temperature History of 7,500-lb Block Retorted During Run 2.

The liquid products from the process were periodically discharged from the oil collection tank, sampled, and analyzed.

All of the gaseous product from the retort that was not recycled was metered and flared. The stack gas stream was sampled every 2 hours for analysis by gas chromatography. For ease of operation two chromatographs were used: One to separate and determine carbon dioxide and the higher hydrocarbons, and the other to determine carbon monoxide, oxygen, nitrogen, and methane.

A water-cooled ring, located directly above the grate, is used to cool the exposed carbon steel surfaces in the lower part of the retort. Heat flux transducers are used to determine heat losses from the retort vessel.

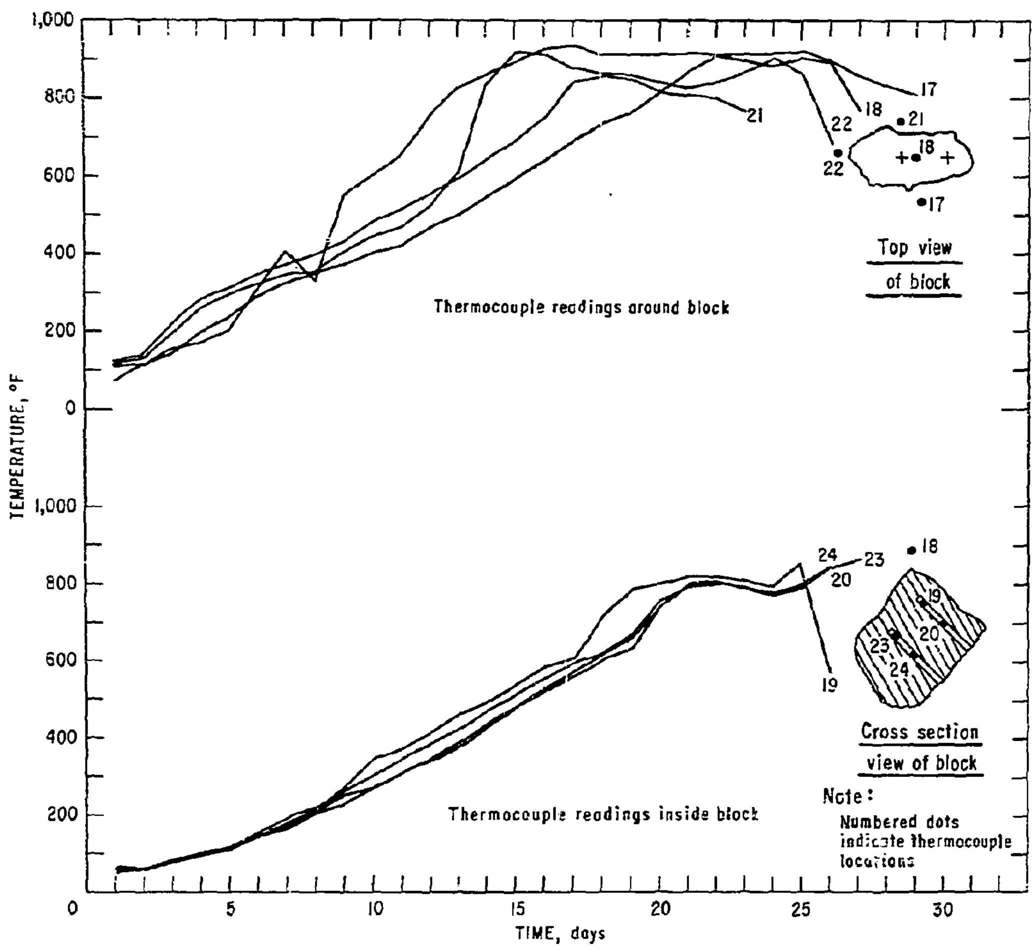

FIGURE 6. - Temperature History of 10,100-lb Block Retorted During Run 3.

RESULTS AND DISCUSSION

The two runs covered in this report are part of a series of nine studying the effect of oxygen content in the retorting gas and the space velocity of the retorting gas through the shale bed. Recycle gas and air are combined to obtain the desired oxygen content of the retorting gas entering the retort.

Table 1 lists the summary data for runs 2 and 3. For run 2, the oxygen content of the retorting gas averaged 14.5 percent with a space velocity of 1.94 $ft^3/ft^2/min$. For run 3, the oxygen content of the retorting gas averaged 9.2 percent with a space velocity of 1.24 $ft^3/ft^2/min$. Results of the two runs were considerably different. For example, run 2 required only about

12 days to complete, and the oil recovery was 62.2 percent of Fischer assay; run 3 lasted for 52 days, and an oil recovery of only 39.3 percent was obtained. Maximum bed temperatures observed for runs 2 and 3 were about 1,600° and 1,000° F, respectively. The oil from run 2 has a higher specific gravity, pour point, viscosity, and nitrogen content and a lower sulfur content than the oil from run 3. The spent shale analyses show zero oil content for run 2 and 0.4 gallon of oil per ton for run 3.

TABLE 1. - Summary data for runs 2 and 3, 150-ton retort

Run number	2	3
Date	3/23-4/4/70	6/24-8/15/70
Length of run, days	12.25	52.0
Operating conditions:		
Shale charge, tons	178.67	179.37
Retort pressure, psig	3.0	3.0
Time burner on, hours	3.0	4.0
Burner heat release rate, Btu/hr	633,000	475,000
Air rate (dry), scfm	135	39
Do, scfh	8,100	2,340
Do, scf/ton shale	13,300	16,310
Avg air temp into retort, ° F	28	74
Recycle gas rate (dry), scfm	67	90.3
Do, scfh	4,000	5,420
Do, scf/ton shale	6,600	37,680
Avg recycle gas temp into retort, ° F	43	82
Oxygen content of retorting gas, pct	14.5	9.2
Space velocity, ft^3 gas/ft^2 bed/min	1.94	1.24
Stack gas rate (dry), scfm	177	41
Do, scfh	10,600	2,460
Do, scf/ton shale	17,400	17,110
Avg stack gas temp, ° F	81	117
Gas produced in retort (dry), scfm	42	2
Do, scfh	2,500	120
Do, scf/ton shale	4,100	830
Max retort differential press, in. H_2O	0.6	0.4
Avg barometric press, in. Hg	22.93	23.22
Avg ambient temp, ° F	26	69
Avg retorting advance rate, in/hr	1.75	0.41
Max bed temp, ° F	1,600	1,000
Bed compaction, pct of initial height	5.6	6.8
Oil shale properties:		
Fischer assay, gal/ton	25.4	24.7
Oil content, wt pct	9.6	9.5
Water content, gal/ton	2.9	2.6
Bulk density, lb/ft^3	80.0	80.3
Mineral carbon, wt pct	4.78	4.71
Mineral carbonate, wt pct	23.90	23.54
Ash, wt pct	68.73	68.79
Organic carbon, wt pct	11.04	11.17
Hydrogen, wt pct	1.66	1.69

TABLE 1. - Summary data for runs 2 and 3, 150-ton retort--Continued

Run number	2	3
Oil shale properties--Continued:		
Nitrogen....wt pct..	0.38	0.37
Sulfur....wt pct..	0.57	0.58
Gross heating value....Btu/lb..	2,267	2,293
Recovery:		
Oil....lb..	21,280	12,990
Do....gal..	2,830	1,740
Water....lb..	16,030	21,540
Do....gal..	1,924	2,586
Spent shale....tons..	125.86	158.43
Oil recovery....vol pct of Fischer assay..	62.2	39.3
Oil properties:		
Distillation, vol pct:		
IBP-392° F at 760 mm Hg....	5.7	6.0
302°-392° F at 40 mm Hg....	25.8	27.2
437°-522° F at 40 mm Hg....	42.4	40.9
Residuum....	26.1	25.9
Specific gravity, 60°/60° F....	0.903	0.896
Gravity....° API..	25.2	26.4
Pour point....° F..	70	50
Viscosity....SUS at 100° F..	79	64
Hydrogen....wt pct..	11.76	12.11
Nitrogen....wt pct..	1.77	1.45
Sulfur....wt pct..	0.76	1.10
Carbon....wt pct..	84.58	84.63
Ash....wt pct..	0.01	0.01
Gross heating value....Btu/lb..	18,660	18,810
Stack gas properties (dry basis):		
H_2....vol pct..	0.06	0.03
O_2....vol pct..	1.31	4.07
N_2....vol pct..	66.39	78.53
CO_2....vol pct..	28.67	15.37
CO....vol pct..	2.32	0.90
CH_4....vol pct..	0.69	0.68
C_2H_6....vol pct..	0.35	0.27
Higher hydrocarbons....vol pct..	0.21	0.16
Specific gravity, air = 1....	1.31	1.06
Spent shale properties:		
Fischer assay....gal/ton..	0	0.4
Oil content....wt pct..	0	0.2
Water content....wt pct..	0.28	1.7
Hydrogen....wt pct..	0.14	0.43
Mineral carbon....wt pct..	1.83	4.70
Organic carbon....wt pct..	1.24	3.10
Mineral carbonate....wt pct..	9.15	23.50
Ash....wt pct..	91.91	78.24
Nitrogen....wt pct..	0.10	0.19
Sulfur....wt pct..	0.56	0.51
Gross heating value....Btu/lb..	117	356

Figure 4 is an example of the temperature profiles observed in the shale bed. For run 2, the bed temperature rise from 200° F to the maximum of 1,200° F required about 4 days, whereas in run 3 the temperature rise from 200° F to the maximum of 900° F required about 8 days. In addition, the temperature spread indicated by the eight thermocouples was more uniform for run 2 than run 3.

Figure 5 shows the temperature history of the block weighing 7,500 lb retorted during run 2. The temperatures inside the block rose rapidly, from about 100° to 1,200° F in less than 5 hours, and the maximum temperature of about 1,600° F was reached about a day later. The thermocouples located around the block also indicated a rapid temperature rise. These temperature data indicated that the block heated as fast or perhaps even faster than the surrounding shale bed.

After the retort cooled, about 7 feet or 40 tons of spent shale were dug out from above and surrounding the large block to expose it in place. The block was still intact, but numerous cracks had developed. Samples from the center of the block analyzed zero oil content.

Figure 6 shows the temperature history of the 10,100-lb block retorted during run 3. Again, the thermocouples inside the block indicated a

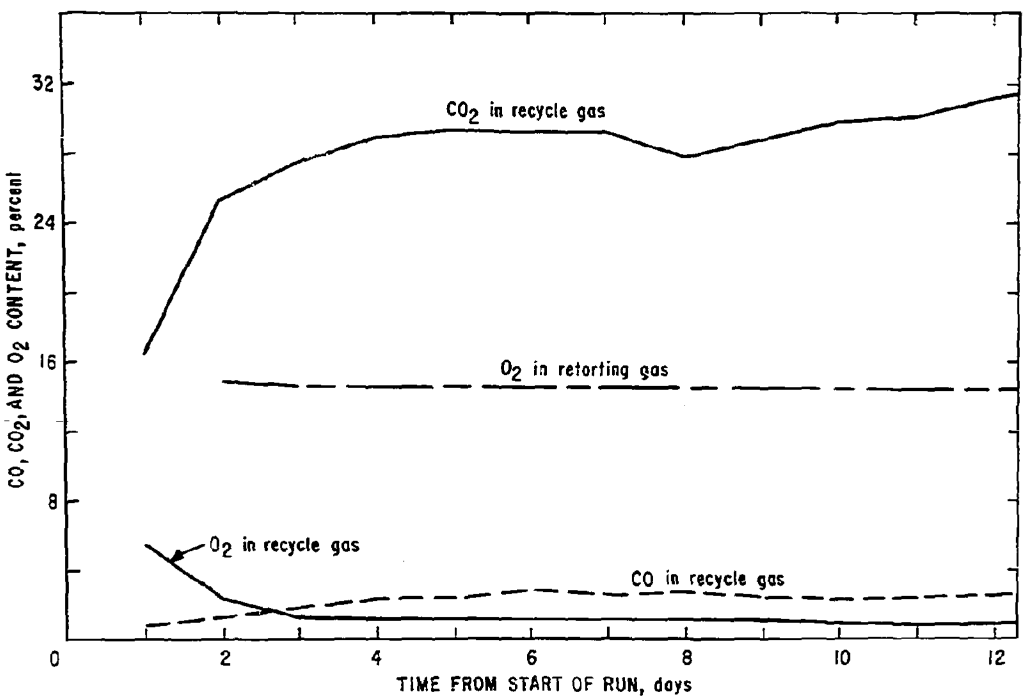

FIGURE 7. - Selected Gas Analyses for Run 2.

temperature rise similar to that in the surrounding shale bed. About 15 days were required to heat the block from 200° to the maximum of 800° F.

Figure 7 shows some gas analyses from run 2. Recycle gas and air rates were set to maintain an oxygen content in the retorting gas of about 14 percent. Oxygen, carbon dioxide, and carbon monoxide values of the recycle gas are also shown. The oxygen content in the recycle gas averaged 1.31 percent and was quite uniform after about 2 days.

Figure 8 shows some gas analyses from run 3. For this run, air and recycle gas rates were set so that an oxygen content of 7.0 percent in the retorting gas would have been realized if the oxygen content in the recycle gas had been near 1 percent as in run 2. However, the oxygen content in the recycle gas, averaging 4.07 percent, ranged from 11.8 to 1.7 percent. This resulted in a higher than expected average oxygen content of 9.2 percent in the retorting gas. The carbon dioxide content of the recycle gas reached a maximum of about 19 percent, somewhat lower than in run 2 because of the relatively low decomposition of mineral matter at the lower retorting temperatures of run 3. Figure 8 also shows that more than half the run had been completed before equilibrium conditions were reached.

Table 2 lists the material balances and tables 3 and 4 list the carbon and energy balances, respectively, for each run. Although none of these

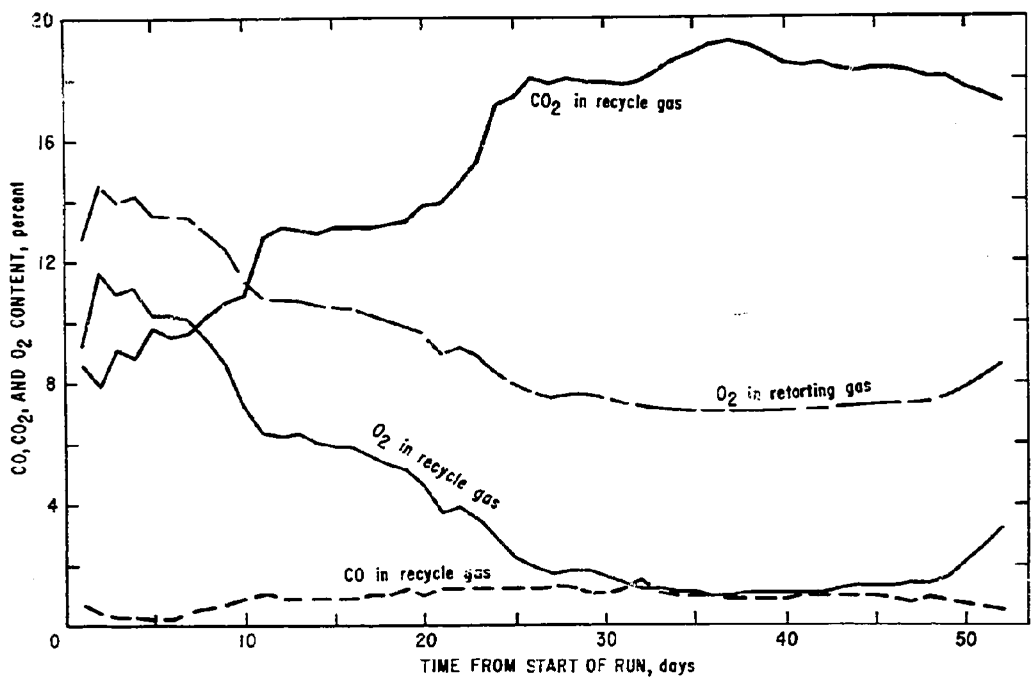

FIGURE 8. - Selected Gas Analyses for Run 3.

balances close precisely, the indicated gains and losses are not considered abnormal for work of the scale and nature.

TABLE 2. - Material balances for runs 2 and 3, 150-ton retort

	Pounds	Percent
Run 2:		
Material in:		
Oil shale	357,340	66.1
Natural gas	70	-
Air (wet)	182,900	33.9
Total in	540,310	100.0
Material out:		
Spent shale	251,710	45.5
Shale oil (dry)	21,280	3.9
Stack gas (dry)	257,200	46.5
Water:		
In oil	8,070	-
In stack gas	6,520	-
From heat exchanger	7,960	-
Total water	22,550	4.1
Total out	552,740	100.0
Excess out	-	2.3
Run 3:		
Material in:		
Oil shale	358,740	61.4
Natural gas	80	-
Air (wet)	225,640	38.6
Total in	584,460	100.0
Material out:		
Spent shale	316,870	52.5
Shale oil (dry)	12,990	2.1
Stack gas (dry)	247,060	41.0
Water:		
In oil	16,010	-
In stack gas	4,930	-
From heat exchanger	5,530	-
Total water	26,470	4.4
Total out	603,390	100.0
Excess out	-	3.2

TABLE 3. - Carbon balances for runs 2 and 3, 150-ton retort

	Pounds	Percent
Run 2:		
Carbon in:		
Oil shale	56,530	99.9
Natural gas	50	0.1
Total in	56,580	100.0
Carbon out:		
Spent shale	7,730	13.5
Oil	18,000	31.4
Stack gas	31,540	55.1
Total out	57,270	100.0
Excess out	-	1.2
Run 3:		
Carbon in:		
Oil shale	56,970	99.9
Natural gas	60	0.1
Total in	57,030	100.0
Carbon out:		
Spent shale	24,720	46.2
Oil	10,990	20.6
Stack gas	17,730	33.2
Total out	53,440	100.0
Loss	-	6.3

TABLE 4. - Energy balances for runs 2 and 3, 150-ton retort

	Btu/ton of raw shale	
	Run 2	Run 3
Heat in:		
Enthalpy of raw shale	-56,000	-34,000
Enthalpy of air	-11,929	-888
Enthalpy of recycle gas	-4,464	3,620
Heating value of raw shale	4,534,000	4,586,000
Heating value of recycle gas	142,049	615,988
Heating value of ignition gas	9,196	10,677
Total heat in	4,612,852	5,181,397
Heat out:		
Enthalpy of spent shale	149,338	143,088
Enthalpy of off gas	17,999	29,047
Enthalpy of oil	2,378	889
Heat of vaporization, uncondensed water	111,853	130,897
Enthalpy of condensed water	4,037	3,364
Heating value of spent shale	166,245	628,880
Heating value of off gas	432,786	882,642
Heating value of oil	2,223,426	1,361,655
Energy removed from grate cooling ring	15,678	289,429
Heat losses to atmosphere	452,042	1,719,413
Carbonate decomposition	331,462	52,893
Total heat out	3,907,244	5,242,197
Unaccountable loss....percent..	15.3	-
Unaccountable gain....do....	-	1.2

Note: All heating values are gross; reference temperature is 77° F; gas data are based on standard of 60° F and 1 atmosphere.

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