OIL SHALE RETORTING UNDER ADIABATIC CONDITIONS

V.V. Mahajan, R.E. Lumpkin, F.M. Gragg, R.J. Fraser
Occidental Research Corporation
1855 Carrion Road
La Verne, California 91750

Abstract

A small pilot plant has been designed and constructed to simulate the retorting of oil shale under conditions pertinent to a modified vertical in-situ shale oil recovery process. The pilot plant consists of a 6-inch diameter (15.2 cm) fixed bed reactor, 4 feet (1.2 m) long with provision for heat supply along the wall. This external heat supply balances the heat losses so that the retort operates like an in-situ retort. Results from typical pilot plant runs are included. These runs are characterized by high oil yields and excellent mass, energy and heat balance closures.

Introduction

Oil shale reserves in the western United States constitute one of the most promising future domestic sources for liquid fuels. Many processes have been proposed to tap this resource, and a number of above ground and in-situ methods have been tested on a pilot scale. Occidental Petroleum Corporation has developed a process, combining features of both true in-situ and above ground processing, called the vertical, modified in-situ retorting process.

Since mid-1972, Occidental has formed and burned four in-situ retorts, and a fifth will be ignited this month. Processing results (McCarthy and Cha 1976; Fernandes and Cha 1977) and plans for commercial development (Sass and Lumpkin 1977; Ashland and Occidental 1977) have been published previously. This paper discusses one aspect of oil shale research being conducted by Occidental Research Corporation (ORC) in support of the continuing field tests.

ORC, the central research division of Occidental Petroleum, was responsible for the initial development of the Oxy oil shale process. In January 1975, Occidental Oil Shale, Inc. was formed to continue the development; ORC retained responsibility for medium and long-range supporting research. A major facet of ORC's extensive oil shale program has been the design, construction and operation of small-scale, batch retorts capable of simulating in-situ processing.

This paper briefly reviews the history of batch retorting experiments, explains the rationale behind the ORC design, and presents some of the experimental results.

Early Work on Batch Retorting

Occidental's vertical, modified in-situ process involves preparing a packed bed of shale underground and retorting this bed in place to produce the oil. The retorting process is somewhat similar to the N-T-U process, studied by the Bureau of Mines, for above ground batch retorting of oil shale (Ruark and others 1956). The Bureau constructed two identical 40-ton (36.2 metric ton) retorts, shaped like inverted truncated cones, 16 feet (4.8 m) high, with internal diameters of 8 2/3 feet (2.6 m) at the top and 10 1/3 feet (3.1 m) at the bottom. Experiments in these retorts proved that a combustion front can be propagated through a bed of oil shale particles, and the recovery of oil from such a unit could be a self-sustaining operation. Useful information was obtained on the effects (on retorting) of air flux, air to recycle gas ratio, grade of shale and particle size of shale.

A 10-ton (9 metric ton) retort was constructed by the Bureau of Mines, at what is now the Laramie Energy Research Center (LERC) of ERDA, specifically to simulate in-situ retorting. This retort was built to study the processing of shale ungraded in size and varying in oil content, and to examine slow retorting of large shale particles. The work was generally successful. A number of reports have been published (Carpenter and Sohns 1974; Dockter and others 1972). Maximum recovery of oil in this retort was approximately 52 percent of Fischer assay at a superficial gas velocity of 4 SCFM/ft^2 and an oxygen concentration of 16 percent by volume in the inlet gas. Low- to medium-grade shale, containing 10-25 gal (0.04-0.11 m^3)/ton oil, was used in most of the runs in this retort.

LERC researchers have also operated a bigger retort, with a nominal capacity of 150 tons (135 metric tons), using shale in chunks as large as 4 feet (1.2 m) across (Harak and others 1970; Harak and others 1974). Maximum oil recovery was 65.8 percent of Fischer assay at a superficial gas velocity of 1.25 SCFM/ft^2 and an oxygen concentration of 15 percent by volume. The Fischer assay oil content of the shale charged was in the 20-30 gal (0.08-0.12 m^3)/ton range.

Some similarities have been observed in the operation of these two LERC retorts, such as the linear dependence of the retorting rate on the oxygen flux (Carpenter and Sohns 1974). However, a recent attempt (Dockter and Harak 1976) to correlate results from these two units led to the conclusion that they have different operating characteristics. A major reason for the observed discrepancies appears to be heat losses of unknown magnitude in these two vessels. Estimates of these losses range from 25-40 percent of the total heat content of the shale bed. Such losses are likely to distort the retorting behavior and limit the applicability of the data.

Other investigations, less extensive than those in the big retorts at LERC but employing batch processing with significant heat losses at the walls, have been reported by researchers at Sandia (Arnold 1975); LERC (Wise, Miller and George 1976); Phillips Petroleum (Needham and others 1976); the University of California (Branch and Ness 1976); and the University of New Mexico (Nuttall 1976). Operating small scale units in this manner complicates the analysis of the data and makes the extension of the work to larger retorts difficult. Proper modelling can be used to predict the behavior of retorts with non-adiabatic walls, but using data from such an experiment to confirm or evaluate the model is very risky.

Rationale for Adiabatic Operation

It is usually possible to derive many different process models for a system as complex as in-situ oil shale retorting. Normally, the simplest model chosen for use is the one that satisfactorily predicts the system's behavior over the expected range of operating conditions. It is frequently very difficult to determine acceptable level of simplicity (or complexity) because mechanisms that are important in laboratory or pilot plant work may not be important in the field. It is therefore desirable to avoid making analysis of small-scale experimental data any more difficult than is inherent in the process. Allowing heat losses through the walls of a batch retort seriously complicates the necessary model. Radial temperature profiles become significant and a two-dimensional, rather than a one-dimensional, treatment of the process is required. A two-dimensional model can be avoided by proper experimental design.

Two laboratories, other than Occidental Research, have reported data from retorts constructed to eliminate, or at least control, heat losses through the vessel walls. A small retort has been built at Laramie (Duvall and Jensen 1975), with a series of separately controlled, six-inch (15.2 cm) long heaters over its 13-foot (3.9 m) length. The vessel is three inches (7.6 cm) in diameter, with a one-inch (2.5 cm) pipe down the center; the shale is placed in the annulus, which is only one inch (2.5 cm) across. Only limited data have been published, and, if adiabatic operation has been attempted, it has not yet been reported.

Lawrence Livermore Laboratory (LLL) has constructed two retorts, one with a capacity of 6 tons (5.4 metric tons) and the other capable of holding 275 lbs (125 kg) of shale (Ackerman and Sandholtz 1976). This work also appears to be promising, but the published data are not sufficient to evaluate how well the systems work in controlling heat losses.

Occidental developed a mathematical model for in-situ oil shale retorting (McCarthy and Cha 1976) and used this model in the design of the early field experiments. Parameters in the model were primarily derived from Laramie data. Subsequently, we refined the model by using results from the field tests, but felt that additional information would help to extend and improve it. Field work is notoriously difficult to instrument and analyze properly, and the tests are so expensive and time-consuming that the effects of many variables cannot be adequately explored. Consequently, ORC decided that a pilot retort, two feet (0.6 m) in diameter and 10 feet (3.0 m) long, holding approximately one ton (.9 metric ton) of shale, was large enough to provide a good simulation and small enough to permit many runs within a short span of time. ORC began preliminary design of this retort in late 1974.

Occidental's Pilot Retort (1 ton; .9 metric ton)

We decided to build a retort with heavily insulated walls rather than resort to heaters to control the wall temperature and prevent heat loss. This route seemed to offer ease of operation and lower cost. Construction was started in May 1975 and completed in November. The first run was made during the last week of November; the seventh and last run was completed in March 1976. Analysis of temperature profiles observed during the seven runs indicated that almost half of the heat generated at the burn front was used to raise the temperature of the refractory lining of the retort. This loss imposed severe radial temperature profiles and made it difficult, if not impossible, to extrapolate the results to the behavior of in-situ retorts. We concluded that heaters would be required to generate meaningful data.

The features of our first pilot retort vessel are shown in figure 1. There are three layers of insulation. The inner layer is a hardened fire brick, 4 1/2 inches (11.4 cm) thick, backed by insulating brick of equal thickness, and a 1-inch (2.5 cm) layer of fiber insulation. Since the surface temperature of the steel shell never rose above ambient temperature, the insulation was quite effective in preventing loss of heat from the vessel. Nonetheless, a significant quantity of heat was required to raise the temperature of the refractory lining as the flame front passed through.

A radial temperature profile from the second run is shown in figure 2. Temperatures were measured by moving a thermocouple across the radius and therefore are corrected for the flame front movement. The temperature falls over $800^{O}F$ ($302.4^{O}C$) from the center of the retort to the wall. Most of the heat removed from the bed stays within the fire brick, and, as the flame front passes, it flows back into the shale. Thus, little heat is lost to the atmosphere but the refractory lining distorts the process results by acting as a heat storage device.

Significant improvements to a number of the peripheral systems were made during the seven runs. Flow control and oil collection systems were developed that satisfied all of the requirements for good mass

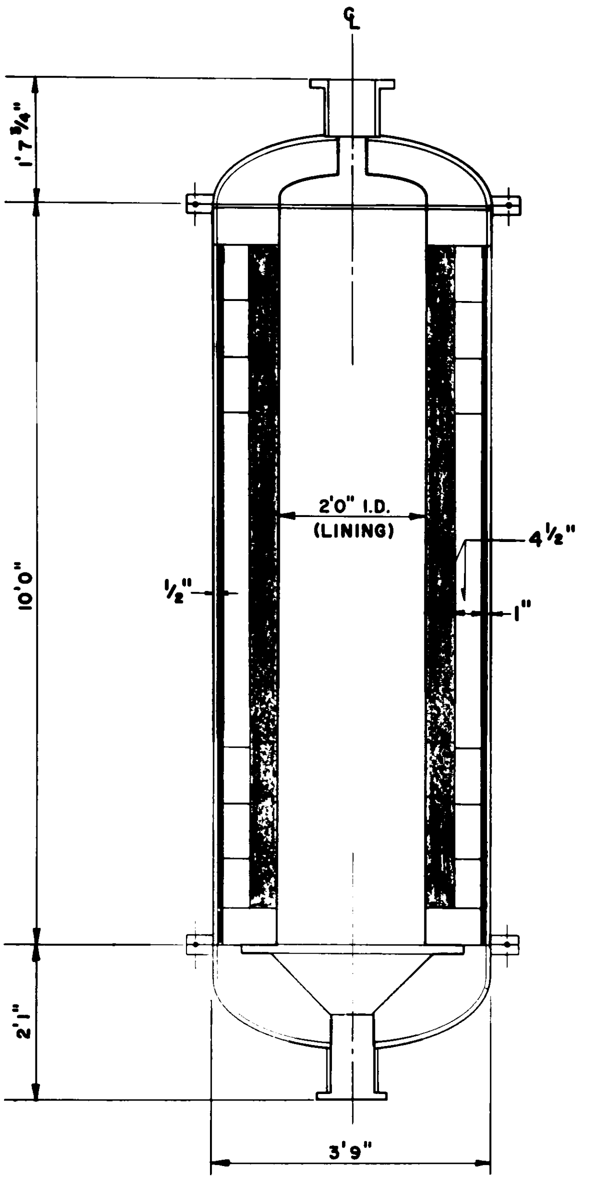

Figure 1. - Schematic diagram of 1-ton retort.

balances and steady operation. The gas chromatographic analysis of gas exiting from the retort was fine-tuned; a system for data collection and logging was developed. Data from this unit were recorded on magnetic tape cassettes for later playback onto time-shared computers. The software for collating and manipulating such data was also developed during these runs.

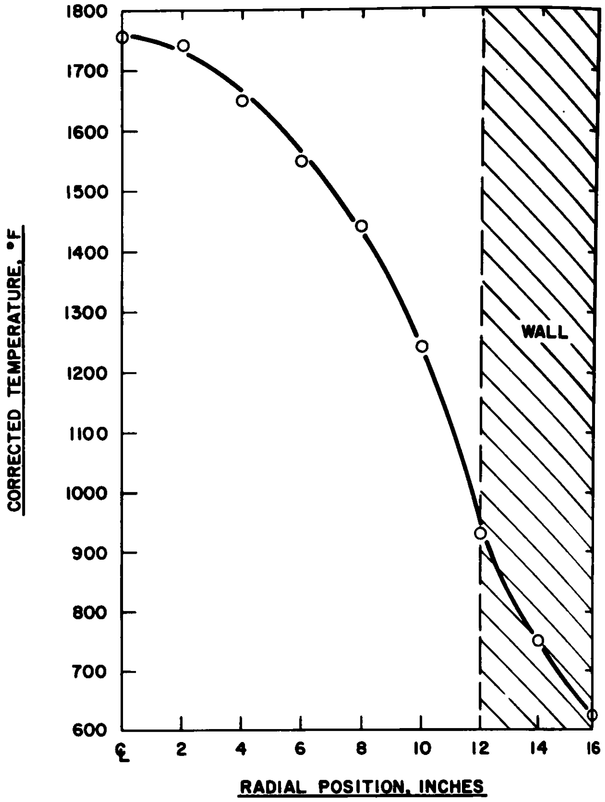

Figure 2. - Radial temperature profile, 1-ton retort run 2. T.C. 1-K-18.

The "Adiabatic" Mini-Retort

A decision was made in mid-January 1976 to construct an "adiabatic" pilot plant capable of accurately simulating in-situ retorting behavior. The design incorporated a series of separately-controlled heating sections to maintain the reactor skin temperature equal to the center line temperature. Ideally, no heat would be added to the shale bed in the retort, only enough energy to balance heat losses. The reactor was to be six inches (15.2 cm) in diameter and four feet (3.1 m) long, with 16 separate heating sections, each 3 inches (7.6 cm) high.

We recognized one limitation of this equipment at the time of its design: particle sizes would probably be restricted to less than 1/2 inch (12.7 mm). The 1-ton (.9 metric ton) pilot retort would have to be rebuilt to examine the behavior of large rocks. The new unit had its own advantages, however. Construction would be faster and cheaper; time required to perfect the operation and achieve temperature control would be reduced. In addition, turnaround time would be shorter; more runs could be made within a given time span.

The "adiabatic" retort assembly is shown schematically in figure 3. Total length of the retort vessel is 7 feet (2.1 m). The top and bottom portions, 18 (45.7 cm) and 12 (30.5 cm) inches long, respectively, are heated to control the temperatures of the inlet and exit gases. A gas preheater, provided at the top, doubles as a steam generator. Gas flows are metered both into and out of the retort; provision is made for recycling the gas exiting from the retort. Part of this is diverted through an oxygen meter and then to the gas chromatograph. The oxygen meter is used to monitor bed ignition which is achieved through the use of a Calrod coil heater.

Construction of this pilot plant was completed on schedule at the end of April. Tests without shale occupied the first week of May; the first retorting run was made May 9-10, 1976. The mass balance for this run closed to within 1 percent. Unfortunately, it took until the 19th run on November 10 before the heat and energy balances

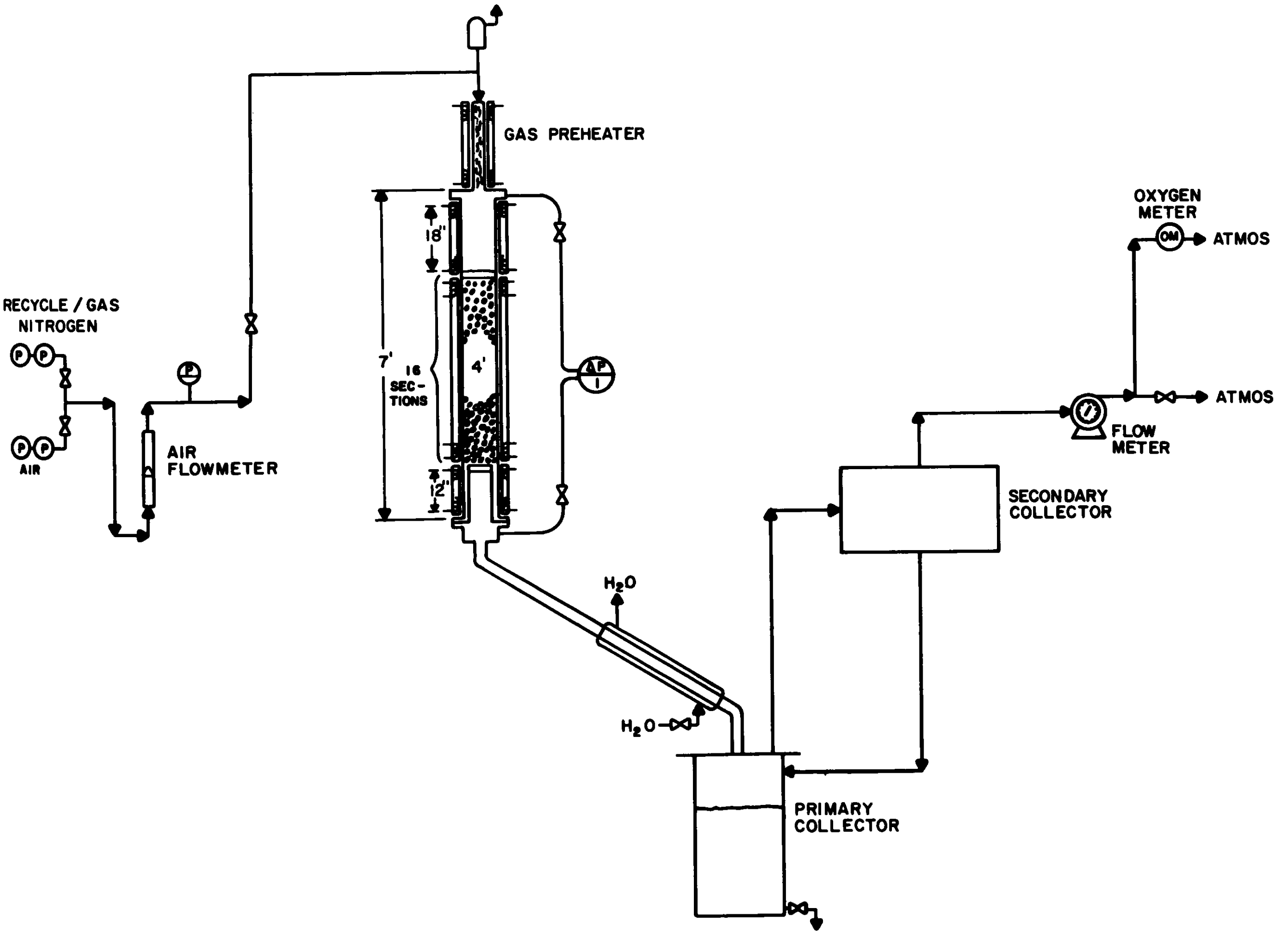

Figure 3. - Schematic Diagram of the Adiabatic Pilot Retort.

closed satisfactorily and the other criteria for adiabatic operation were met.

The effects of heat loss on retort behavior are illustrated by the results plotted in figures 4 and 5. Heat losses, during early runs in the pilot retort, were caused by random periodic malfunctions in the temperature control scheme at various points in the bed. Because of the localized nature of these control problems, overall heat balance closure cannot be used as the only parameter to define the deviation from ideal performance. However, it is still possible to establish qualitative trends from these two graphs. The curves show that air consumption by the process decreases and rate of advance of the flamefront increases as the heat balance closure improves. The three runs, included in these figures, were nominally identical. Only the control techniques were changed. In absence of a similar run, after control techniques were perfected, predictions of the computer model under the same conditions are included for comparison. These predictions seem to be generally consistent with the pattern established by the data.

Five tests were applied to the run data to determine if the system was operating satisfactorily. Our criteria were:

1. Radial temperature profiles for an ideal retort should be flat. During the initial phases of the development of this retort, radial temperature gradients of up to 100°F/inch (37.8°C/2.5 cm) were observed, demonstrating the severity of the heat losses. The worst gradients observed during current operation are less than 5°F/inch.
2. Overall energy balances should close. The early runs showed that 20 to 30 percent of total system energy was lost as heat through the walls. This was not a significant improvement over earlier LERC retorts. Improvements in the design of the mini-retort have led to closures within 5 percent, indicating that losses are minimal.
3. Overall heat balances should close. Heat that flows into the vessel, plus heat generated by exothermic reactions, should equal heat consumed by endothermic reactions plus heat that flows out of the vessel. Early runs showed heat losses of 40 to 50 percent of input; later runs closed to within 10 percent. Greater scatter is expected in these balances than in energy balances because parameters are in-

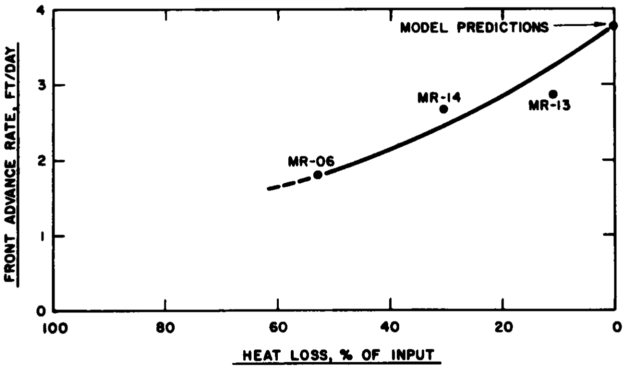

Figure 4. - Retorting rate as a function of heat loss in the mini-retort.

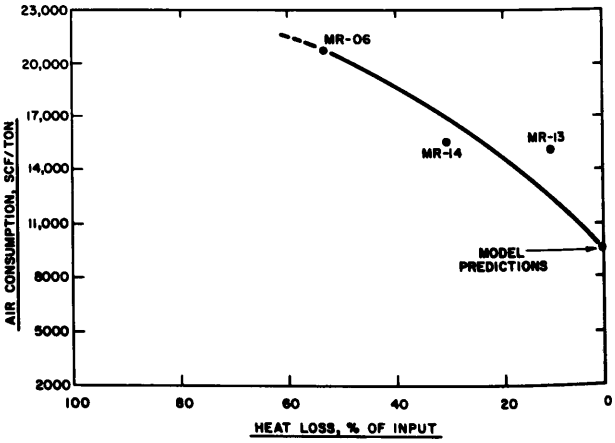

Figure 5. - Air consumption as a function of heat loss in the mini-retort.

volved that are not directly measured. For example, the extent of various reactions as a function of time must be estimated to produce final heat balance.

4. Intermediate energy and heat balances should also close. These balances were not computed in the early phases of the work, but they are now incorporated in the main data analysis program and close to within 10 percent of perfect balance. They require a knowledge of the extent of each of the chemical reactions throughout the bed and accurate values for the oil production and hold-up rates.
5. The combustion front should propagate steadily. The earliest runs in the mini-retort were characterized by an irregular propagation rate of combustion and retorting fronts through the bed. The situation was caused by poor temperature control, and, in fact, some runs were abandoned to prevent damage to the retort. More recent runs have shown that this control problem is solved; retorting and flame fronts move in a steady progression through the shale.

One criterion, avoided in deciding when the retort reached a satisfactory level of operation, was a comparison between experimental results and computer simulation model. Our purpose in building the pilot plant was to test and extend the model, and we felt that it was necessary to establish independent criteria for evaluating effectiveness of our pilot plant temperature control.

Current Operations

Since adiabatic operation was demonstrated, more than 25 additional runs have been made. Two runs per week are routine; occasionally three runs can be made in that time. Data are logged on magnetic tape and fed to a time-shared computer for collation and initial data analysis, a technique developed for the 1-ton (.9 metric ton) pilot plant.

Data for four runs are summarized in table 1. These runs were selected to demonstrate the capabilities of the unit; the data have no other special significance. Two diluents for inlet air were used and two replicate tests made with each gas. The same shale was charged in all runs; inlet oxygen concentration and flow rate were maintained as constant as possible. Oil yields with steam averaged 95 percent; those with nitrogen 92 percent. Retorting rates did not vary significantly. Gas exiting from the retort has a higher heating value when steam is used because one of the diluents (water) is condensed out. Gaseous products from retorting are very similar in all cases on an inert-free basis.

Mass, energy and heat balance closures for all of these runs were satisfactory. Reproducibility was excellent; even though average yields for both diluents are above 90 percent, the difference between diluents is significantly greater than the difference between replicate runs. These yields should only be regarded as proof of experimental precision. Both absolute and relative values for oil yield may change significantly under field operating conditions.

Summary and Conclusions

Occidental Research has built and operated an adiabatic mini-retort for oil shale. Mass, energy and heat balance closures are good; reproducibility of runs and consistency of data appear to be excellent. We believe that this retort can adequately simulate in-situ operation, within limits imposed by the size of the equipment.

We are currently making a series of minor modifications to the retort to streamline our operation. A PDP-11/34 dedicated computer is being installed to improve data handling and analysis. We will then be able to make real-time comparisons between theory and experiment.

Table 1. - Adiabatic mini-retort runs: comparison of effect of diluent gases.

Diluent	Nitrogen		Steam	
Run No.	MR-19	MR-21	MR-20	MR-23
Total inlet gas flow rate SCFM/ft^2	0.733	0.726	0.772	0.718
Air flow rate, SCFM/ft^2	0.471	0.467	0.551	0.485
Oxygen concentration at inlet, mole %	13.50	13.50	14.99	14.17
Oil Yield, % of Fischer assay	91.59	92.73	95.16	94.70
Retorting rate, ft/day	2.52	2.59	2.90	2.39
Air required, SCF/ton shale	9370	9496	9750	9486
Off-gas heating value, BTU/SCF	36	42	55	64
Mass balance closure, %	96.93	97.66	97.09	96.47
Energy balance closure, %	91.05	95.07	94.93	98.84
Heat balance closure, %	92.78	91.03	91.52	91.95

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