MORGANTOWN ENERGY TECHNOLOGY CENTER

DOE/METC/SP-197

TOPICAL REPORT

OIL SHALE

COMBUSTION/RETORTING

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May 1983

UNITED STATES DEPARTMENT OF ENERGY
MORGANTOWN ENERGY TECHNOLOGY CENTER
MORGANTOWN, WEST VIRGINIA

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E R R A T A

DOE/METC/SP-197
TOPICAL REPORT
OIL SHALE COMBUSTION

May 1983

On page 12, second column, last paragraph, " 10^4 C/second," should read:
" 10^4 C/second."

On page 20, the twenty-third item in the first column, "Phosphorus, P_2 ," should read: "Phosphorus, P_2O_5 ."

We apologize for any inconvenience this may have caused.

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MORGANTOWN ENERGY TECHNOLOGY CENTER

TOPICAL REPORT

OIL SHALE COMBUSTION/RETORTING

TECHNOLOGY DEVELOPMENT AND

ENGINEERING DIVISION

UNITED STATES DEPARTMENT OF ENERGY

Morgantown Energy Technology Center

Morgantown, West Virginia 26505

May 1983

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1.0 INTRODUCTION

The oil shale research program at the Morgantown Energy Technology Center (METC) has been directed toward a means of recovering maximum thermal and chemical energy using the fluidized-bed process for retorting and/or combustion of various oil shales with emphasis on eastern and low-grade oil shales. The feasibility studies of low-grade oil shales also led to the development of a number of innovative process design and hardware concepts. METC's experience in oil-shale utilization demonstrated that the fluidized-bed combustion/retorting technology offers attractive features that aid improvements for increased energy recovery and improved shale oil yield.

METC, the U.S. Department of Energy's (DOE) lead center for fluidized-bed combustion research, conducted a number of feasibility studies on the combustion and retorting of five oil shales: Celina (Tennessee), Colorado, Israeli, Moroccan, and Sunbury (Kentucky). These studies generated technical data primarily on (1) the effects of retorting conditions, (2) the combustion characteristics applicable to developing an optimum process design technology, and (3) establishing a data base applicable to oil shales worldwide. During the research program, METC applied the versatile fluidized-bed process to combustion and retorting of various low-grade oil shales. The scope of METC's major activities is summarized in Sections 1.1 through Section 1.3.

1.1 EFFECTS OF RETORTING CONDITIONS

The study focused on five independent variables: (1) retorting temperature, (2) in-bed residence time, (3) shale particle-size distribution, (4) fluidization-gas velocity, and (5) fluidization-gas composition. The results could lead to developing optimum process design and operation for high-quality (maximum similarity to crude oil) hydrocarbon production. The results could also aid in the design of commercially feasible hardware for efficient heat recovery.

1.2 APPLICABLE COMBUSTION CHARACTERISTICS

This study focused on three variables: (1) combustion feasibility/efficiency, (2) flue gas characteristics, and (3) resultant ash characteristics. The resulting technical information will lead to the development of a high, total energy-conversion efficiency for the tested shales. It will also enable the pollutants emit-

ted during the conversion processes to be identified and characterized and the design parameters for the energy conversion process to be determined.

1.3 OIL-SHALE DATA BASE

The technical data base generated by METC during the oil-shale utilization research program could be the means to categorize by composition to (1) draw correlations between U.S. and foreign oil shales; (2) expand the data base of fluidized-bed combustion/retorting for worldwide applicability; and (3) initiate a data base on environmental, health, and safety issues.

2.0 BACKGROUND

METC became involved in oil shale utilization when the State Department requested design information for Israeli oil shale. The general charter of METC included conducting combustion feasibility studies of low-grade fuels.

Subsequent involvement led to the germination and reduction-to-practice of innovative concepts to increase the energy recovery from shale in terms of high liquid yield and maximum thermal energy utilization.

In oil shale utilization, the primary issue is how to extract the maximum energy from the oil shale, in terms of both chemical (hydrocarbons) and thermal energy. Maximum energy recovery is dependent on developing a totally integrated energy-conversion system that produces high quality hydrocarbon yield. Also, a second major issue is the composition of the oil liquid which includes the hydrocarbon and contaminant compositions. These factors determine the compatibility of the oil with available refining processes and various end uses. In addition, the disposal and/or utilization of large amounts of inert spent shale pose other significant questions that affect efficient oil-shale use.

Some earlier work (Smith, 1977) inferred that significant changes in the character of the oil yield occurred with increased shale heating rates. Specifically, these changes were increased total yield, increased aromaticity, and decreased olefin/paraffin contents. Achieving these characteristics appeared to be an attractive goal from both an economic and production standpoint. Fluidized-bed processing of shale offers heating rates and a flexibility in choice of retorting conditions which far exceed that available with conventional packed-bed retorting approaches.

Consequently, fluidized-bed process for retorting of shale was judged a potentially fruitful area for investigation. Fluidized-bed technology features include excellent solids mixing, high-heat transfer rate, high-solids throughput, and simple operation. Therefore, METC used these characteristics to conceptualize innovative oil-shale utilization devices and processes. For example, METC conceived and designed the twin fluidized-bed combustor/retort that successfully and economically recovers the heat of combustion by burning residual carbon and produces clean spent shale for environmentally acceptable disposal. A second device was the pseudo two-stage, fluidized-bed combustor that recovers thermal energy more efficiently by separating devolatilization from the heat-consuming calcination reaction in combustion. METC also developed the use of a porous metal filter to efficiently recover the tiny shale-oil aerosols and demonstrated the use of hot carbon dioxide as a combined shale heating medium and fluidizing gas for improved shale oil yield.

In summary, METC's oil shale utilization experience shows that fluidized-bed combustion/retorting of most oil shales offers a promising method for improved yields and quality. The combustion characteristics also make the process highly applicable for energy uses with fuels of very low heating values, such as the low-grade and hydrogen-deficient oil shales. Moreover, fluidized-bed technology has substantial versatility in achieving maximum energy recovery from oil shale in an efficient and environmentally acceptable manner.

3.0 **PRINCIPAL OBJECTIVES OF FLUIDIZED-BED COMBUSTION AND RETORTING**

METC's efforts have concentrated on identifying and developing process design parameters for high yield and high quality hydrocarbon production and for efficient heat recovery from the shale. Because fluidized-bed technology can handle a wide range of solid, gaseous, and liquid fuels, METC developed the studies discussed in Sections 3.1 through 3.5.

3.1 *DETERMINATION OF RETORTING CONDITION EFFECTS*

Efforts were made to correlate oil yield and composition with various shale types and operating conditions. Also, this study focused on controllable retorting conditions and the effects caused by retorting conditions on shale oil composition. The controllable retorting conditions affect hydrocarbon production as follows:

- Retorting temperature affects the decomposition of kerogen and bitumen into low-molecular weight hydrocarbons. Most oil shales are expected to have an optimum decomposition temperature.
- Residence time of the shale in the retort must be long enough to release the hydrocarbons from the shale. However, if excessive, it causes decomposition or secondary cracking of the retorting products and loss of liquid yield. A low residence time affects the physical size of the retort which, in turn, affects the capital investment.
- Fuel particle size determines the solids heating rate in the retorting unit plus the rate of diffusion of the hydrocarbon vapors from within the oil-shale rock matrix. These factors, likewise, affect the optimum retort size. If the oil shale particle size is reduced too much, an excessive energy consumption will result.
- The fluidization velocity affects the extent and quality of gas-solid mixing and the solids heating rate in the retort. It also affects the vapor residence time in the freeboard region, which, if excessive, causes undesirable secondary cracking of retorting hydrocarbon products. High-fluidization velocity, in contrast, reduces the residence time of the vapor in the freeboard and alleviates the extent of secondary cracking. Excessive fluidization velocity, on the other hand, both aggravates elutriation problems and adds surplus gas to the process that likely will increase the cost and the physical size of downstream vapor handling equipment.
- Fluidization-gas composition affects the physical structure of the rock matrix. It can chemically influence the rock matrix to enhance the retorted hydrocarbon vapors diffusion out of the oil-shale rock matrix. A shorter residence time in the rock matrix reduces the extent of secondary cracking, the carbon deposition, and loss of liquid yield.

These retorting conditions, in turn, affect the following:

3.1.1 Off-Gas Composition and Quantity

The off-gas generally consists of reducing compounds with high-sulfur content. Under certain conditions, the sulfur may cause catastrophic corrosion and, subsequently, material failures of the retorting equipment. Other compounds usually found in the retor-

ting off-gas include water vapor, carbon oxides, hydrocarbons, nitrogen compounds, and trace quantities of catalyst-poisoning (arsenic and phosphorus) compounds.

3.1.2 Total Liquid Yield

The total liquid yield determines the economic feasibility of the retorting process. This, in turn, influences initial investment considerations for any type of commercial venture. However, one major problem exists with the collection of shale oil aerosols formed during the retorting process. Conventional dust particle recovery processes have difficulty in collecting the oil which tends to be in the form of tiny liquid aerosol droplets. Thus, elaborate collection processes must be used. This increases both the capital investment for the equipment and the operational complexity which makes the retorting process less commercially attractive.

3.1.3 Shale-Oil Composition and Physical Characteristics

The compatibility of the product with current refinery technology depends primarily on the hydrocarbon composition and the physical and chemical properties of the liquid produced during the retorting process. Most state-of-the-art refinery systems are not equipped, at present, to directly process shale oil for two reasons. First, the catalytic-poisoning elements commonly found in shale oil, such as arsenic, lead, and other heavy metals, jeopardize the hydrocarbon processing catalysts. Secondly, the environmental need for significantly lower sulfur and nitrogen levels in the shale oil may involve the cost of adding substantial hydrogen to aid pollutant removal.

Likewise, the market desires the production of shale oil that is similar to crude oil to enhance compatibility with existing refinery technology and to supply required product distributions. Generally, the hydrocarbons in oils are classified into paraffinic, olefinic, naphthenic, and aromatic (PONA) groupings. Crude oil has essentially no olefinic compounds in it; however, conventionally retorted shale oil has a significant fraction. The hydrocarbon conversion processes (hydrogenation, cracking, reforming) will benefit from a shale-oil PONA composition tailored for existing refinery technology.

The shale oil composition is a function of many varying factors. In retorting, the rate of heating and the temperature are known factors that affect the

hydrocarbon composition of shale oil products. Research findings by (Smith, 1977) indicate that (1) a low retorting temperature produces shale oil with a high paraffin content, (2) an intermediate temperature results in high olefinic shale oil content, and (3) a high temperature produces shale oil of a high-aromatic hydrocarbon content.

Smith reported the rate of heating also determines the hydrocarbon composition of shale oil. At a heating rate of 1°C/min, the shale oil is principally paraffinic. At a moderate rate below 100°C/min, the shale oil contains more olefinic hydrocarbons. And, at a fast rate of heating (far above 100°C/min), the shale oil is primarily high in aromatic hydrocarbon content.

3.1.4 Spent-Shale Utilization and Disposal

Utilization and disposal of the spent shale involve important economical and environmental considerations in the design of the retorting process. Maximizing the energy recovered from the combustibles in the spent or retorted shale (i.e., residual carbon) maximizes the heat available for retorting, thus enhancing the process. Similarly, maximizing burn-out appears to ease the disposal problem of the large amount of spent solid wastes. The remaining combustibles may aggravate health and safety issues which are influenced by both the appearance of the spent shale and the long-term effects (i.e., leachability) of shale-waste disposal. Lastly, in view of the considerable amount of minerals in the spent shale, the development of alternative processes such as cement manufacturing and mineral reclamation may also enhance the total process attractiveness.

3.1.5 Removal (Retention) of Sulfur and Nitrogen Residuals

The control of known pollutants, particularly sulfur and nitrogen oxides, remains a key issue in the environmental acceptability of the shale oil retorting process. The retorting process generates known pollutants (hydrogen sulfide, ammonia, and amines) and can be a source of their emissions. In addition, subsequent combustion of untreated shale oil would produce excessive yields of sulfur dioxides and nitrogen oxides. Manipulation of the retorting conditions, however, could reduce the production of potential pollutants to a manageable level and assist in minimizing costs for expensive emission control and treatment equipment.

3.1.6 Correlation of Oil-Shale Type to Shale-Oil Composition

The establishment of a data base from the results of experimental retorting studies can lead to a retorting process model that (1) relates oil-shale compositions and physical properties of selected shale samples to other types of shales for a given process and (2) gives a shale-to-shale comparison ensemble. Proper modeling of the retorting process and subsequent development of algorithms will also be effective for evaluating a wide variety of oil shales worldwide.

3.2 COMBUSTION CHARACTERISTICS OF OIL SHALES

Because of the large amount of inert material inherent in the oil shale and the need for retorting heat, excellent thermal integration and maximum utilization of all the potentially available energy is a requirement. Combustion of fresh or spent oil shale for heat generation thus becomes an integral part of oil-shale retorting. The combustion operation involves the direct conversion of the shale's chemical energy into thermal energy. This thermal energy is then the energy source for the oil shale retorting process. Therefore, it was important to determine the combustion characteristics of oil shales considering the following information.

- *Combustion Efficiency.* Combustion efficiency greater than 99 percent conversion is achievable in the fluidized-bed combustion process. The high efficiency is attributed to adequate bed temperature, adequate particle residence time, sufficient air-to-fuel ratio, and high degree of turbulence inside the fluidized bed.
- *Flue Gas Characteristics.* Flue gas characteristics vary according to the types and concentration levels of pollutants in the shale. The unburned combustibles in the flue gas also affect combustion efficiency. Usually the combustion products of sulfur and nitrogen (SO_2 and NO_x) need to meet Environmental Protection Agency (EPA) and local emission standards. Sulfur dioxide emissions are dependent on the relative sulfur and limestone content of the shale. The nitrogen oxide emission is a function of the nitrogen content of the oil shale and the reducing compounds formed during combustion.

- *Ash Characteristics.* Ash characteristics, in terms of composition and physical properties, such as ash-fusion temperature, may impose limitations for combustion and the disposal process.

3.3 PROCESS IMPROVEMENTS AND PERIPHERAL ENHANCEMENTS

The search for process improvements and alternatives for processes integration was a continuous effort. Data gathered during experimental investigations was reviewed continuously to identify potential improvements as well as peripheral enhancements in shale oil upgrading and shale waste utilization.

3.4 PRACTICAL USE OF THE DATA BASE

The experimental results obtained in the retorting and combustion tests of a wide range of oil shales with varying characteristics have been added to the fluidized-bed combustion/retorting data base. The data has been well documented in order to form a basis for improving energy recovery in terms of heat recovery, shale-oil quality, and yield in an environmentally acceptable manner. The properties relevant to different shales can be correlated according to shale types and operating conditions. The environmental, health, and safety data are being collected, reviewed, and documented for future use. Potentially, this data base can quantitatively predict the potential applicability of various shales based upon identifiable characteristics and provide process design and evaluation information.

4.0 MAJOR CHARACTERISTICS OF FLUIDIZED-BED COMBUSTION/RETORTING

Fluidized-bed technology is not a novel process at the refinery level. For the last four decades, U.S. petroleum refineries have used fluidized-bed technology for catalytic petroleum refining processes. Fluidized-bed combustion, an offshoot of fluidized-bed refinery operations, has emerged over the last 20 years as an achievable technology.

4.1 MAJOR COMBUSTION PROCESS FEATURES

The process characteristics of the fluidized-bed process include the following:

- High degree of in-bed mixing of solids and general bed uniformity that results from the turbulent conditions achieved by fluidizing gas agitation.
- Excellent degree of interparticle heat transfer rates that result from the turbulent mixing of solids.
- Extensive thermal inertia due to the large amount of inert solids in the fluidized bed.

The implications of these characteristics on the combustion process can be summarized as follows:

- A wide variety of "low-grade" fuels can be burned because of the vigorous mixing, high-thermal inertia, and high fraction of inert solids normally present.
- In-bed sulfur capture (emissions control) is possible with calcium carbonate/oxide either as a sulfur sorbent as a material addition or as naturally present in the "inert" bed material.
- An inherently low level of nitrogen oxide emission exists due to the low fluid-bed combustion temperature and the presence of reducing compounds in the fluidized bed.
- A high potential for practical and economical applications exists because of inherently high throughput, simple design, and operational characteristics in a device that does not require the additional external pollution control equipment.

4.2 *FEATURES OF FLUIDIZED-BED RETORTING*

The advantages of fluidized-bed process for retorting oil shale are as follows:

- High in-bed heat transfer rate results in very rapid heating of cold, raw shale particles to retorting temperature.
- Large solids throughput is possible because of a high degree of bed uniformity and because the raw shale feedstock can attain the desired retorting temperature without long residence time in the retort.
- Operational flexibility exists because the fluidized bed can handle a wide variety of oil shale types and sizes. For example, the shale fines generated by mining and processing operations tend to block the gas passages of conventional packed-bed retorts but do not hinder fluidized-bed retorts.

- Secondary hydrocarbon cracking resulting from the short residence time of the gaseous retorted hydrocarbon products in the retort (and in the shale matrix) is minimized.
- Economics is improved because the retort is inherently smaller for a given throughput and offers the potential for modular construction. The simplicity of operational design also may produce a long-term savings in operation and maintenance costs.

5.0 **KEY TECHNICAL ISSUES**

Oil shale utilization activities at METC have focused on gaining maximum energy extraction from oil shale; specifically in terms of heat recovery and yield of high quality (aromatic content) shale oil. The three primary technical issues are chemical and thermal energy recovery, shale oil characteristics, and effluent disposal.

5.1 *CHEMICAL AND THERMAL ENERGY RECOVERY*

Energy must be efficiently and nearly totally extracted from oil shale in both chemical (hydrocarbon) and thermal forms to obtain a desirable process.

- *Hydrocarbon.* Chemical energy is recovered in the form of product shale oil and noncondensable retorting gases. Of major significance are the selection of fluidization process parameters, the fluidizing-gas composition, integration of the non-condensable gases, and the techniques used for recovery of shale oil aerosols.
- *Thermal.* The thermal energy extraction (sensible heat) generally is used to provide heat for the retorting process. Of major significance are the use of hot spent-shale solids, the reduction of residual carbon in the spent shale, and the control of heat-consuming carbonate calcination reactions.

5.2 *SHALE OIL CHARACTERISTICS*

Shale oil is a crude feedstock that must be readily convertible into products which possess characteristics that meet market demand. The type and composition of hydrocarbons in the shale oil, as well as trace elements, determine refinery compatibility, technology, and costs. The characteristics that are of specific interest are as follows:

- *Hydrocarbon Types.* The hydrocarbon type and composition in the shale oil determine the kind and extent of the refining process. Generally, the classification of the hydrocarbon types include (1) saturated (paraffinics and naphthenics), (2) unsaturated (olefinics), and (3) aromatics. For comparison normal crude oils are extremely low in olefin content.
- *Sulfur/Nitrogen Contents.* These elements are primarily important due to both the effect they have on the refinery costs and equipment (hydrogen demand and catalyst life particularly). Equally important is that sulfur and nitrogen content in the refined combustion fuels will generate known pollutants (sulfur dioxide and nitrogen oxides).

In summary, refineries must be capable of processing the product shale oil. The compatibility of shale oil with the local refinery depends on several variables: the arsenic/nitrogen/sulfur contents, the PONA distribution, the physical properties, and the specific chemical compositions. The retorting end product is preferred to be a group of hydrocarbons that will be compatible with available refinery processes and systems.

5.3 ENVIRONMENTAL (EFFLUENT DISPOSAL)

The large amounts of inert materials in oil shale and the particle growth during retorting result in a disposal problem for a high volume of solid waste. The various potential environmental and economic considerations relating to the disposal techniques are important concerns for solid effluent disposal.

6.0 TECHNICAL RESULTS

Fluidized-bed process variables can be controlled to a significant degree to accommodate shale of widely variable composition and influence the liquid yield character and quantity. Identification of the material-process-yield relationships is a critical factor in determining the necessary conditions for maximizing energy recovery and the production of useful products. The scoping studies and experimental efforts described summarize the specific areas of investigation by METC to identify these critical factors. Descriptions of the fluidized-bed units used in these investigations are included in the Appendix for reference.

6.1 SHALE-OIL COMPOSITION AND PRODUCT CHARACTERISTICS

Product characteristics are determined by the nature and composition of the oil shale and processing conditions acting on the shale. In a conventional oil refinery, the oil from oil shale retorting is refined to produce products which have a wide spectrum of hydrocarbons with various molecular structures and weights. The conventional petroleum refinery is designed to separate and upgrade the hydrocarbon products. Usually, about half of petroleum crude will be converted to motor fuel, mainly gasoline. The rest will be converted into kerosene, diesel fuel, naptha, petrochemical feedstock, fuel oils, and heavy fractions. For gasoline with improved antiknocking properties and petrochemical feedstocks, aromatics are favored.

In most conventional packed-bed oil shale retorting operations, large lumps of shale are piled together and contacted with hot retorting gas in either a countercurrent or cocurrent manner. The shale temperature rise rate is restricted by heat transfer limitations and is typically in the 1° to 10°C/min range. This relatively slow rate was shown (Smith, 1977) to form a considerable amount of straight chain and/or saturated hydrocarbons.

METC performed various shale retorting tests using the test configuration design briefly described in Appendix B and collected the product oils. Celina, Sunbury, Colorado, Moroccan, and Israeli shales were retorted and the product oils characterized. The hydrocarbon compositions found were plotted in a ternary diagram (see Figure 1) according to (1) saturated (paraffinic and naphthenic), (2) unsaturated (olefinics), and (3) aromatic hydrocarbon fraction.

From experience in retorting of Colorado oil shale, the rate of heating influences the types of hydrocarbons formed. For example:

- At a shale heating rate of approximately 1°C/min, the oil composition consists mostly of paraffinic hydrocarbons. This is representative of most fixed or in situ retorting processes.
- At a shale heating rate in the range of 10° to 100°C/min, the principal hydrocarbons are mostly olefinics.
- At a shale particle heating rate significantly above 100°C/min, shale oil consists mostly of aromatic hydrocarbons.

FIGURE 1: PONA DISTRIBUTION OF LIQUIDS PRODUCED BY FLUIDIZED-BED RETORTING

Based on these results, a fast shale heating rate would appear to be favored for retorting, especially in the case of hydrogen-deficient oil shales. The inherently high degree of solids mixing makes this fluidized-bed retorting approach very desirable for producing a highly-aromatic product from hydrogen-deficient oil shales. Also, it appears applicable to a wide spectrum of shale "richness," i.e., Colorado to Celina (Tennessee) shale.

6.2 SENSITIVITY TO RETORTING CONDITIONS

Exploratory research indicated that fluidized-bed retorting would adapt to processing oil shale fines, allow high-solids throughput, be less prone to solid-handling problems, and provide more efficient inter-particle heat transfer than the conventional packed-bed, moving-bed or in situ retorting processes.

The fluidized bed is readily adaptable for heating oil shales and can result in high oil yields for hydrogen-deficient oil shale. Hydrogen combines with carbon on a one-to-one ratio to form aromatic hydrocarbons. This reduces the chance of forming residual carbon. Preliminary investigations indicated the fluidized bed increases shale oil yield and produces a product high in aromatic content.

Parametric studies were conducted at METC to determine the significance of process variables that affect oil yields from various oil shales and to determine optimum operation for maximizing oil yield.

The retorting studies were conducted in 2-inch diameter, electrically heated, fluidized-bed retort equipment. The equipment consisted of cyclones, a glass-wool filter, and a porous-metal filter to recover the oil shale aerosols and, subsequently, to collect the shale oil liquid. An illustration and description of the units and operating capability are presented in Appendix B.

The process variables are (1) retorting temperatures and feed rates, (2) residence time and particle size, (3) fluidization-gas velocity, and (4) fluidization-gas composition. The results obtained from laboratory investigations of each of these process variables are described in Sections 6.2.1 through 6.2.4.

6.2.1 Retorting Temperature and Feed Rates

The retorting temperature is an important process variable affecting the shale-oil yield and composition. Published information (Smith, 1977) also indicates

that (1) low-retorting temperature favors paraffinic hydrocarbon production, (2) intermediate temperature produces more olefins, and (3) high temperature favors aromatics production. The formation of aromatics also theoretically increases oil yield for oil shales because of the generally low hydrogen-to-carbon ratio (as compared to crude oil).

There are a number of incentives for employing low-temperature retorting. In addition to lower fuel costs incurred when operating at lower temperatures, shale oil yield actually may be increased with a lower retorting temperature. Excessive temperature tends to cause cracking of the released hydrocarbons within the retort. The by-products of hydrocarbon cracking include free carbon and a considerable amount of light hydrocarbons, which are difficult and generally impractical to condense. The uncondensable hydrocarbon gases and carbon deposition represents substantial oil yield loss. Also, carbon deposition can result in significant operation problems at high oil shale retorting temperatures. In addition, there is the obvious benefit of lower fuel costs when combustion temperatures are reduced.

As METC gained experience and became more familiar with different types of oil shales, tests were conducted with oil shales of different mineral composition and crystal formations. Two very dissimilar oil shales were chosen from those available to investigate the sensitivity of the retorting temperature on the basic shale type. Israeli oil shale was selected for its low porosity and the Indiana oil for its high silica content. Both shale sizes were in the range of 30 x 45 mesh and were tested using a fluidizing nitrogen gas velocity in the range of 22.4 cm/sec (about 1.8 to 2.0 times the minimum fluidization velocity) with a residence time between 22 to 31 minutes. The results are shown in Figure 2 for Indiana oil shale and Figure 3 for Israeli oil shale. According to the data, both oil shales showed a surprisingly similar retorting temperature profile with maximum yield points of approximately 900°F. It should be noted that not all the oil shales have this identical optimum retorting temperature and feed rate (in-bed residence time).

Feed rate of oil shale was the subject of interest in another series of tests. From a commercial point of view, short average solid residence time reflects a high-solid throughput, and consequently, low-capital investment cost. It is of economical interest to explore the optimum solid feed rates for maximum oil yield.

FIGURE 2: THE EFFECT OF BED TEMPERATURE ON OIL YIELD, INDIANA OIL SHALE

ISRAELI OIL SHALE
30 x 45 MESH
FLUIDIZING VELOCITY: 22.4 cm/sec

$\mu = U/U_{mf} = 2$
RESIDENCE TIME: 22 - 24 MIN.
FISCHER ASSAY: 5.4% (wt)

FIGURE 3: THE EFFECT OF RETORTING TEMPERATURE ON OIL YIELD, ISRAELI OIL SHALE

To determine the combined effects and best combination of retorting temperature and feed rate for high quality oil yield, METC designed a series of retorting tests. A 2-inch diameter, electrically heated, fluidized bed (see Appendix B) was used in this experiment. The oil shale used, supplied by the U.S. Shale Corporation, was mined from Celina, TN. Oil shale distribution was 30 x 45 mesh and the fluidization velocity used was 27.4 cm/sec. Retorting temperature versus feed rate results are represented in a contour plot (see Figure 4) where the oil yield weight percentage is also shown as a third parameter. A regression analysis was applied to correlate the oil yield (Y) as a function of retorting temperature (T) and the oil shale feed rate (F). The regression analysis produced the following relationship:

$$Y = -24.814 + 0.0536T - 2.703 \times 10^{-5}T^2 \\ + 0.381F - 8.96 \times 10^{-5}F^2 \\ - 1.799 \times 10^{-5}T \cdot F$$

Application of the regression formula indicates the most favored retorting temperature is 910°F with a feed rate of 180 gm/hr in the 2-inch diameter electrically heated fluidized-bed retort. This result is also evident on the contour plot.

Other factors that influence the oil yield are secondary cracking and collection efficiency. In general, the cracking within the fluidized bed may be catalyzed by the shale rock matrix. The cracking of hydrocarbon vapors in the freeboard is due to excessive gas temperature. Whereas the freeboard cracking is common to all the hydrocarbons produced from oil shales, the catalytic cracking cannot be ascertained without the rock matrix being clearly defined. Collection efficiency of shale oil aerosols can also affect the oil yield. The collection efficiency is a function of aerosol size, population, flow pattern, and a host of physical and chemical characteristics of the collection system.

FIGURE 4: CONTOUR PLOT OF OIL YIELD AS A FUNCTION OF
FEED RATE AND RETORTING TEMPERATURE

6.2.2 Residence Time and Particle Size

There are three residence times that are critical to shale oil yield: (1) solid in-bed residence time, (2) retorted hydrocarbon in-rock residence time, and (3) freeboard hydrocarbon vapor residence time.

Solid in-bed residence time is determined by the size of the retorting vessel. For a given retort volume, the solid residence time is inversely proportional to the feed rate.

Two major considerations with regard to the solid in-bed residence time of a particular oil shale are the solid residence time and the particle size which influences the rate of hydrocarbon migration out of the shale rock matrix. Solid residence time is dictated by the thermal inertia of the fluidized bed and rate of feed. A small volume of bed material at temperature will be quickly lowered if the rate of feed is greatly increased. Also, with the bed volume held constant, high solid throughput has the effect of reducing residence time. Therefore, it is desirable to maintain a balance between solid in-bed inventory and shale feed rate for a particular size shale.

The in-rock hydrocarbon residence time is determined by the oil shale particle size, porosity, and shale-rock formation and heating rate. During retorting, the hydrocarbon vapors formed from the decomposition of kerogen and vaporization/cracking of bitumen, the degree of thermal cracking, and the catalytic cracking is a function of the shale-rock matrix and specific physical dimensions of the particular shale. The shale rock matrix also may contribute to a number of catalytic activities in hydrocarbon conversion. It is not exactly clear how the catalytic functions take place or what the effect is on shale oil yield and composition. However, it appears advisable to achieve a physical separation of the retorting products from the shale rock matrix at the first available opportunity because of the likelihood of catalyzed hydrocarbon cracking.

Thermal cracking in the freeboard may also contribute to the low shale oil yield by the formation of hard-to-condense gaseous products. For most fluidized-bed solid processing vessels, a considerable freeboard height, known as the Transported Disengaging Height (TDH), insures adequate separation of solid particles from elutriating gas-solid streams. TDH is defined as the freeboard height at which the particle dust loading (concentration) approaches a constant value above which the particle dust loading remains invariant. For a given fluidized

bed, the TDH is a function of bed diameter, fluidization gas velocity, and the in-bed geometrical configuration. General design practice of fluidized bed calls for a freeboard height no less than the TDH. In addition to the freeboard functioning as a settling chamber, in fluidized-bed combustors the freeboard also burns off the combustibles coming off the fluidized bed. Thus, to insure the fulfillment of these functions, most fluidized-bed designs incorporated a high freeboard. In the case of fluidized-bed retorting of oil shales, long freeboard residence time may not be desirable. And thus, could lead to secondary cracking of oil shale vapors in the freeboard.

Low-porosity Israeli oil shale was selected for a series of experiments to determine the effect of particle size and residence time on oil yield. A tightly compacted oil shale (low porosity) tends to restrict the diffusion of the shale oil vapors from the rock matrix during retorting. This causes a prolonged in-rock residence time for shale oil vapors and corresponds directly to the shale particle size. Three batches of Israeli oil shales with differing sizes were used: (1) 20 x 45 mesh with an average diameter of 0.50 mm, (2) 20 x 30 mesh with an average diameter of 0.72 mm, and (3) 14 x 20 mesh with an average diameter of 1.13 mm.

All three batches were retorted at 900°F. This temperature was used because previous experiments identified 900°F as optimum retorting temperature for Israeli oil shale.

Basically, the retorting process is affected by the temperature of the retorting bed. From a heat conduction point of view, heating of oil shale particles is very rapid, usually less than 1 minute. It can be seen from Figure 5 that retorting took less than 20 minutes for the Israeli oil shale with a size of 30 x 45 mesh. By comparison, to reach a plateau of oil yield, the 20 x 30 mesh cut required more than 20 minutes and the 14 x 20 mesh cut took approximately 25 minutes to reach an equilibrium. The results (see Figure 5) indicate that an in-bed residence time of less than 25 minutes will be adequate for low porosity shales.

Note that the solid in-bed residence time should not be confused with the heating rate. The solid residence time is the average solid residence time in the retort. Residence time is a function of the feeding system and solid-flow pattern. Inside a fluidized bed, the vigorous particle-to-particle heat transfer rate will heat the shale particle at a rate of approximately 104°C/second.

FIGURE 5: EFFECT OF PARTICLE SIZE AND RESIDENCE TIME ON OIL YIELD

6.2.3 Fluidizing Gas Velocity

The fluidization velocity determines the state of turbulence and vapor residence times within the fluidized bed and the freeboard. Generally, the fluidization-gas velocity should be low enough that its volume is an acceptable burden on the downstream shale oil recovery system. Yet, it must be high enough to generate sufficient in-bed mixing and heat transfer rates and to sweep away the retorting products without appreciable secondary cracking. To insure good fluidization, the fluidizing gas velocity is usually no more than twice the minimum fluidization-gas velocity.

Velocity of the fluidizing gas must be maintained above a minimum required to suspend the in-bed solid particles in a "fluid" state but not so high as to create large gas bubbles.

When fluidized at a gas velocity above that required for minimum fluidization, the gas will pass through the fluidized bed in the form of increased bubble size. In many ways the bubbles are the major locomotion of solid-to-solid heat and mass transfer. However, large bubbles induced by excessive fluidization-gas velocity are a major cause of shale particle attrition especially from the gas jets existing inside the bed near the distributor region. Fluidization-gas velocity determines the gas in-bed residence time and also the gas/vapor freeboard residence time.

For most oil shale retort operations, the freeboard temperature remains hot enough to cause secondary cracking of hydrocarbon vapors. The cracking reaction reduces the shale oil yield when some of the hydrocarbon compounds convert to carbon deposits and hard-to-condense light hydrocarbons. Minimizing the freeboard residence time by increasing the

fluidization gas velocity will reduce the extent of the secondary cracking.

Figure 6 shows the results of tests to determine the effect of fluidization gas velocity on the oil yield of the Israeli oil shale. The Israeli oil shale was selected because of its tight rock formation. The particular shale used had a size cut of 30 x 45 mesh and was retorted at a minimum fluidization velocity of approximately 10.0 cm/sec and then at the increasingly higher velocities. The retorting temperature was 900°F. The results shown are for two tests; in Figure 6 solid in-bed residence time was 10 minutes and 20 to 40 minutes for the other test. It is of interest to note that regardless of the solid in-bed residence time, the optimum fluidization-gas velocity should be on the order of twice the minimum fluidization velocity. This velocity seems adequate for most oil shales.

6.2.4 Fluidizing-Gas Composition

Fluidizing-gas composition is believed to play a significant role in oil-shale retorting. The use of carbon dioxide, either alone or as an enriching agent in the fluidizing gas, was investigated because conceptually it offered two major advantages:

- CO₂ has the potential to increase bed particle porosity and reduce the hydrocarbon residence time within the shale rock matrix.
- CO₂ has the potential to provide some of the heat for retorting by recirculating some of the hot flue gas from the combustor. Thus, the cost (energy loss) of the retorting process is reduced through large-scale integration of the system's operations.

FIGURE 6: EFFECT OF FLUIDIZING-GAS VELOCITY ON OIL YIELD

The effect of significant carbon dioxide partial pressure on enhancing pore openings in dolomite, (a high magnesium-carbonate mineral) for sulfur retention in pressurized fluidized-bed combustion has been well documented. The effect is to reduce the vapor residence inside the rock matrix, lessen the tendency for the organics to carbonize, and lessen the risks of secondary cracking of the hydrocarbons.

A test was designed to determine the yield improvement which may be expected with carbon dioxide additions to the fluidizing gas. The oil shale was retorted at 900°F with a fluidizing velocity about 1.4 times that of the minimum fluidization velocity. Solid in-bed residence time for the tests was approximately 25 minutes. Data from five tests (see Figure 7) show an oil yield increase from 7.53 percent to 8.25 percent as the proportion of carbon dioxide was increased from 0 to 100 percent. Thus, it was concluded that for western oil shale, the carbon dioxide indeed enhances the shale oil yield to an appreciable extent.

6.3 COMBUSTION CHARACTERISTICS

The retorting process is endothermic and, therefore, combustion of a fraction of the shale hydrocarbon content is generally the most practical means of supplying the required heat. Since the retorting processes do not entirely consume all the hydrocarbons in the shale, both "fresh" and "spent" (retorted) shale can be considered as potential combustion fuels.

As mentioned earlier, the fluidized-bed combustion process is nearly ideal for this very low heating value "fuel." METC examined the combustion characteristics of the various types of oil shales in a number of feasibility studies using the fluidized-bed combustion process in a manner which could be related to the retorting process. The fluidized bed handles two different kinds of fuel, fresh and spent shale. At present, the experimental efforts have been restricted to fresh-shale combustion. The study of spent shale was limited to analysis only.

FIGURE 7: THE EFFECT OF COMPONENTS AND CONCENTRATIONS OF FLUIDIZING GAS ON THE OIL YIELD EFFICIENCY

The combustion characteristics of oil shales are also important because, for environmental reasons, the presence of sulfur and nitrogen compounds in the oil shales may form the toxicants, sulfur dioxide (SO_2) and nitrogen oxides (NO_x). The combustion of some oil shale, by virtue of their high limestone content, can reduce sulfur-dioxide emissions to an environmentally acceptable level. Other reducing substances generated or released by combustion, most notably residual carbon with a porous surface, can lessen the emission of nitrogen oxides. Additionally, the presence of trace elements in the oil shale, such as arsenic and other heavy metals, is a critical consideration in the characterization of oil shales for fluidized-bed combustion/retorting. Disposal of large volumes of spent shale containing residuals/leachable compounds may create environmental health and safety issues and, thus, require additional research to reduce the hazard.

6.3.1 Fresh Shale

METC has successfully employed the fluidization process for the combustion of low-grade and/or eastern oil shales. The fluidization process, owing to its inherent ability of vigorous solid mixing, offers many advantages over the conventional combustion process. For example, the intense combustion in the fluidized bed permits the use of low-grade fuels rich in ash such as the low-grade and eastern shales. The objectives of experiments on fluidized-bed combustion of low-grade and/or eastern oil shales include:

- Determine the feasibility of direct combustion of low-grade and/or eastern oil shales in an atmospheric fluidized-bed combustor.
- Determine the combustion and operational characteristics that may be unique to these oil shales.

Table 1 shows a typical ultimate analysis of the five different oil shales used in the experimental runs. It is worth noting that all the tested oil shales have over 50 percent ash content. Among the five shales, Tennessee shale contains the highest ash (83.2 percent) and sulfur (2.84 percent). In addition, it has the lowest carbon (7.82 percent). Elemental-ash analysis of the tested oil shales are given in Table 2. The Israeli, Moroccan, and Colorado oil shales contain a considerable amount of calcium oxide in the ash. During the combustion of these oil shales, no limestone was required to meet the EPA sulfur dioxide emission limits. On the other hand, both eastern oil shales contain less than 1 percent of calcium oxide in the ash.

Table 3 lists the heating value of the tested shales. Among the five types of oil shales, the Tennessee oil shale has the lowest heating value. This shale's heating value is slightly over 1,600 Btu/lb which is about one-eighth of bituminous coal. However, no combustion instability was encountered throughout the combustion test in the fluidized-bed combustor.

TABLE 1. ULTIMATE ANALYSIS OF TESTED OIL SHALES

TYPE OF OIL SHALE ULTIMATE ANALYSIS, WT%	COLORADO OIL SHALE	TENNESSEE OIL SHALE	KENTUCKY OIL SHALE	ISRAELI OIL SHALE	MOROCCAN OIL SHALE
Moisture	0.58	1.15	0.97	5.14	4.38
Ash	68.05	83.20	76.52	56.61	62.49
Sulfur	0.70	2.84	2.30	2.15	2.14
Hydrogen	2.20	1.17	1.85	1.52	1.62
Nitrogen	0.74	0.33	0.56	0.42	0.40
Carbon	17.65	7.82	16.17	14.86	16.23
Oxygen	10.07	3.49	1.55	19.30	12.74

TABLE 2. ELEMENTAL ANALYSIS OF ASH OF TEST OIL SHALES

TYPE OF OIL SHALE ELEMENTAL ANALYSIS, WT %	COLORADO OIL SHALE	TENNESSEE OIL SHALE	KENTUCKY OIL SHALE	ISRAELI OIL SHALE	MOROCCAN OIL SHALE
Silicon, SiO_2	49.48	59.76	69.21	22.78	39.47
Aluminum, Al_2O_3	11.56	21.79	15.99	10.30	10.83
Iron, Fe_2O_3	5.07	10.49	7.04	3.91	4.82
Calcium, CaO	17.50	0.29	0.84	50.62	30.74
Magnesium, MgO	7.34	1.37	1.36	0.36	5.81
Sodium, Na_2O	4.40	0.52	0.10	1.19	0.10
Potassium, K_2O	2.46	3.27	4.66	0.72	1.48
Phosphorus, P_2O_5	0.45	0.22	0.25	2.86	3.01
Titanium, TiO_2	0.45	0.92	0.91	0.41	0.49
Sulfur, SO_3	2.80	0.36	0.42	6.08	4.42

TABLE 3. HEATING VALUE OF TESTED OIL SHALES

TYPE OF OIL SHALE HEATING VALUE	COLORADO OIL SHALE	TENNESSEE OIL SHALE	KENTUCKY OIL SHALE	ISRAELI OIL SHALE	MOROCCAN OIL SHALE
Heating Value, Btu/lb	2950	1632	3249	1914	2378

Typical experimental results for the combustion feasibility study of the five different low-grade and eastern oil shales are given in Table 4. The most striking results were the two extreme cases on sulfur dioxide emissions measured in the effluent stream during combustion without the addition of any sulfur sorbent. For the eastern oil shales, the sulfur dioxide emission was well above the EPA emission limit of 1.2 pounds of sulfur dioxide per million Btu heat input. For the western shale and the two foreign oil shales, sulfur dioxide emissions are well below the EPA limit.

The nitrogen content of the tested low-grade and eastern oil shales ranged from 0.33 to 0.74 weight percent (see Table 1). This is less than bituminous coal which is slightly above 1.0 weight percent. With the exception of Colorado oil shale, results of the experimental measurements on nitric oxide emissions ranged from a low of 300 ppm for Colorado oil shale to a high of 1,300 ppm for the Kentucky Sunbury oil shale. These values are considerably higher than the NO concentrations measured for bituminous coal

(400 ppm). Factors which are considered to account for these results are:

- The solid-carbon concentration is relatively lower both in the bed and in the freeboard.
- The bed and freeboard temperatures are comparatively lower which affect the NO reduction reaction by carbonaceous solids.

It has been demonstrated that NO emission can be substantially reduced by air staging; i.e., injection of secondary air into the freeboard. A test was carried out on the 6-inch diameter fluidized-bed combustor. The NO concentration of 1,300 ppm was initially measured in the effluent stream as the fluidized bed was operated at 1,400°F. With this bed temperature maintained, about 50 scfh of secondary air was injected into the freeboard and caused the carbon monoxide to be reduced from its initial value of 0.35 to 0.05 percent. This concept of staged combustion is an effective means of control which can be readily incorporated in the design of a fluidized-bed combustion system using oil shales.

TABLE 4. TYPICAL EXPERIMENTAL RESULTS OF FLUIDIZED-BED COMBUSTION OF OIL SHALES

PROCESS VARIABLES	TYPE OF OIL SHALES	COLORADO OIL SHALES	TENNESSEE OIL SHALES	KENTUCKY OIL SHALES	ISRAELI OIL SHALES	MOROCCAN OIL SHALES
OPERATING CONDITIONS						
Bed Temperature (°F)		1550	1530	1660	1300	1450
Superficial Velocity (ft/sec)		3.68	2.10	2.18	2.68	3.96
Static Bed Depths (in)		6	6	6	6	4
Excess Air (%)		33.50	53.39	58.76	44.20	40.0
EXPERIMENTAL RESULTS						
Combustion Efficiency (%)		99.54	98.37	99.39	99.86	97.91
SO ₂ Emission (lb/10 ⁶ Btu)		0.37	24.32	8.80	0.26	0.29
NO _x Emission (lb/10 ⁶ Btu)		0.53	1.67	1.20	1.00	1.00

6.3.2 Spent Shale (Analysis Only)

The ultimate and elemental analysis of fresh and spent shales from retorting experiments is presented in Table 5. From this data, the following observations were made:

- As discussed previously, the presence of carbonates of calcium and magnesium tend to increase the pore size in the oil shale. Therefore, the release of hydrocarbons during oil-shale retorting is made easier. Examination of eastern shales (Celina and Sunbury oil shales low in carbonate content) indicates that a large percentage of carbons was retained by the spent shale. This is reflected in the relatively high heating values of the spent (retorted) shales. Israeli and Colorado oil shales show low heating values for their respective spent shales. The heating value of the Moroccan spent shales is not given because sample size was not sufficient to verify accuracy of this data.
- The formation of silicates and oxides is believed to plug up the pores and veins inside the shale and thereby retard the migration of hydrocarbons to the exterior of the shale. Prolonged residence time of the hydrocarbons inside the rock matrix tends to increase carbon deposition and secondary cracking. The temperature of oil shale retorting is much lower than that of fluidized-bed combustion (1,000°F versus 1,550°F). The silicates formed at the lower temperature may simply block the pores and veins and reduce the migration of hydrocarbons from the rock matrix. This is one theory that may account for the high amount of retained carbon in the spent shale from the retorting process. But, no confirming tests have been conducted.
- The high limestone content of Colorado, Israeli, and Moroccan oil shales retained a large percentage of sulfur in the spent shale. This is expected since the reaction of sulfur compounds with limestone will lead to the formation of calcium sulfides. The extent of the silicates of alkali metals formation reactions are currently being investigated. Under the conditions of oil-shale retorting in this series of tests, no significant amount of silicates of calcium was formed.

6.4 POTENTIAL ENHANCEMENTS

While using the fluidized-bed combustion retorting units, a number of innovative process and hardware concepts were conceived. The innovative concepts addressed the integration of process systems for maximum thermal energy efficiency and the recovery of high-quality hydrocarbons. These innovative concepts are described in Sections 6.4.1 through 6.4.3.

6.4.1 Twin Fluid-Bed Retort Combustor

Retorting oil shale requires heat. And, to achieve a desirable process, the heat must come from a low cost and readily available source such as an unrecoverable combustible in the spent shale. Effectively using these heat sources generally requires a large degree of thermal and mechanical process integration. METC conceived the twin fluid-bed retort/combustor (TFBRC) to achieve this degree of integration with the design simplicity required for reliable operation and low cost.

The key to the TFBRC is its totally integrated thermal system and controlled solids flow path. The design of the TFBRC (see Figure 8) is basically a large fluidized bed with some of the in-bed region and all of the freeboard region separated by a baffle plate between two semi-independent beds. The result is a bed section which burns the retorted spent shale to develop the sensible heat from the residual carbon heat of combustion. The other bed section retorts the raw shale by mixing it with hot, burned spent shale. The hydrocarbon vapors from the retort section are cooled and condensed to recover the hydrocarbons (shale oil). The combustion section's hot flue gas may be sent to a waste-heat boiler for additional heat recovery.

As shown in Figure 8, the combustion and retorting sections of the TFBRC are partitioned by a wall with two interconnected openings for solids to circulate between the two beds. The primary function of the partition is to prevent the cross-contamination of the effluent gases from the retorting and combustion sections. The circulation of beds is maintained by the use of one or more mechanisms; e.g., slanted fluidizing-gas nozzles, differences in fluidized-bed densities (controlled by different fluidizing gas velocities), and a sloped distributor. Raw shale particles are fed to the retort bed section and mixed with hot spent-shale particles coming from the combustion section. The raw shale heats rapidly due to the

TABLE 5. PROPERTIES OF THE OIL SHALES

TYPE OF OIL SHALE/ ANALYSIS ITEMS	U.S. OIL SHALE						MOROCCAN OIL SHALE		ISRAELI OIL SHALE	
	U.S. OIL SHALE CELINA, TN		SUNBURY OIL SHALE, KY		COLORADO OIL SHALE					
	Fresh	Spent	Fresh	Spent	Fresh	Spent	Fresh	Spent	Fresh	Spent
Gallon/Ton Oil	8.0		16.4		23.0		17.7		13.6	
Water	2.4		6.5		3.4		10.2		10.1	
Wt % Oil	3.1		6.5		8.7		7.1		5.4	
Water	1.0		2.7		1.4		4.2		4.2	
Spent Shale	93.1		96.1		87.5		85.0		86.8	
Gas + Loss	2.8		2.8		2.9		3.6		3.5	
Specific Gravity 60°/60°F	0.935		0.945		0.911		0.970		0.963	
Tend to Coke	None		None		Slight		None		None	
Moisture	0.45	0.25	0.93	0.48	0.21	0.14	4.34	8.32	4.4	0.56
Ash	79.37	85.68	75.45	86.79	67.77	79.04	62.77	75.96	64.24	70.92
Hydrogen/Carbon	8.85		9.17		9.52		10.42		9.9	
Hydrogen	1.31	0.40	1.73	0.66	1.74	0.36	1.76	1.27	1.53	0.68
Total Carbon	11.49	7.73	16.94	8.38	16.53	7.82	18.34	7.52	15.22	6.71
Calcium, % of Shale	8.47		0.30		10.1		12.15		23.84	
Nitrogen	0.36	0.70	0.63		0.36	0.15	0.51	0.35	0.35	0.23
Oxygen	0.04		3.52	1.96	12.94	11.96	10.45	13.8	11.45	18.93
Silicon, SiO ₂	60.74	61.84	67.34	66.29	47.56	47.02	40.62	40.64	23.49	24.33
Aluminum, Al ₂ O ₃	13.76	13.76	15.99	15.50	10.07	9.83	11.28	11.29	9.67	9.69
Iron, Fe ₂ O ₃	15.30	15.29	7.22	7.20	4.79	4.56	5.76	5.61	4.24	4.16
Magnesium, MgO	1.52	1.56	1.68	1.36	8.39	7.99	6.43	6.07	0.75	1.31
Sodium, Na ₂ O	0.67	1.09	1.14	1.53	1.18	3.07	0.50	1.01	0.66	1.08
Potassium, K ₂ O	4.91	4.88	4.60	4.65	3.05	2.83	1.61	1.62	0.75	0.76
Phosphorus, P ₂	0.17	0.18	0.28	0.24	0.36	0.38	1.84	1.81	2.31	2.29
Titanium, TiO ₂	0.88	0.86	0.92	0.91	0.44	0.45	0.68	0.69	0.42	0.46
Sulfur, SO ₃	1.01	0.76	0.52	0.43	2.89	2.36	4.88	4.10	6.02	4.46
Heating Value, Btu/lb	2,617	1,699	3,120	1,558	2,296	310	2,543		1,699	486
Ash Fusion, °F	2,133						2,136		2,665	
	2,205						2,160		2,680	
	2,245						2,200		2,695

FIGURE 8: TWIN FLUID-BED RETORT/COMBUSTION

high in-bed solids heat transfer rate and yields hydrocarbon vapors. The continuous solids circulation moves the spent shale from the retorting bed section to the combustion bed section. In the combustion bed section, the residual carbonaceous materials are burned and the hot solids used for retorting are produced. Also, a small fraction of the spent-shale is "bled-off" and balanced with raw shale feed to control the total shale material inventory.

METC constructed a cold model of this concept to study solids circulation. METC also constructed a 9-inch diameter functional model twin-bed retort/combustor to study the actual process phenomena. Shakedown and basic process feasibility testing have been successfully accomplished in the 9-inch hot model. However, more work remains to be done.

Table 6 summarizes typical twin-bed retort/combustor hot model operating conditions. Test results were compromised by gas leaks between the bed sections due to thermal distortion in the partition. The results were affected by some cross-contamination of the product gas streams; i.e., loss of some hydrocarbons generated by the retorting process due to burning. In spite of the cross-contamination, the oil yield

has been above 60 percent Fischer assay. Other than using water-cooled condensation for shale oil collection, no elaborate provisions were made to collect all the shale oil.

In addition to improving process efficiency, the twin-bed concept may offer an environmental benefit. The spent-shale from conventional surface retorting contains an amount of organic residuals which are regarded as potential retardants to revegetation if the spent shale is used as land fill. The twin-bed concept reduces the organic residual materials in the spent-bed shale thus, reducing the environmental impact.

6.4.2 Pseudo Two-Stage Combustion

Many low-grade shales have a substantial amount of limestone. Indiscriminate combustion of the oil shales causes the heat-consuming calcination of limestone into lime. Figure 9 shows the fraction of carbonate decomposition (calcination) as a function of bed temperature. The calcination reaction represents a consumption of 700 Btu/lb and representative shale could contain more than 50 percent calcium carbonate and have a heating value of 1800 Btu/lb. Consequently, combustion of the shale at a temperature high enough to calcine all of the calcium

TABLE 6. PRELIMINARY OPERATING CONDITIONS OF TWIN-BED RETORT/COMBUSTOR

VARIABLES	RETORT	COMBUSTOR
Feed Rate	Nitrogen: 240 scfh Shale: 9.0 Kg/hr	Air: 275 scfh Propane: 5.5 scfh
Preheating Temperature	650°F	
Bed Temperature	880°F 920°F	1360°F - 1370°F
Bed Height	10 inches	10 inches
Gas Composition*		
H/C		1000 ppm ²
O ₂	1.0%	1.8%
CO ₂	3.0%	16.0%
Oil Shale Fischer Assay	8.7% by wt. (Colorado)	
Percent Fischer Assay	61.3	
Bed Material		
Total Carbons		1.91%
H/C		0.02%

FIGURE 9: CARBONATE DECOMPOSITION AS A FUNCTION OF BED TEMPERATURE

carbonate would result in an effective net heating value of 400 Btu/lb. Hence, METC proposed the pseudo two-stage combustion unit. This unit separates the over-bed combustion of dust and volatiles from the fluidized bed in the in-bed combustion process. The pseudo two-stage combustion concept is based on the need for the over-bed and in-bed combustion reactors to take place at different temperatures.

In the pseudo two-stage combustion (see Figure 10) the over-bed region (freeboard) is designed to allow the dust cloud emanating from the bed to be mixed with injected secondary air high over the bed. This allows the volatiles and dust from the oil shale to be burned in the freeboard without causing a significant heat input back into the bed. The dust cloud plus the presence of a vertical baffle act as a screen over the fluidized bed. Thus, a significant temperature difference between the fluidized bed and the freeboard region (e.g., 900° and 1,500°F, respectively) can be maintained. This achieves the equivalent of a two-

staged combustor in a single vessel. In practice, the two-stage system is best achieved as a fluidized-bed combustor with a large or diverted freeboard with the injection of secondary air to support combustion of elutriated dust and fines in the freeboard. The gaseous products of combustion (flue gas) then pass through the convection section and the dust-cleanup system before they are vented into the atmosphere. Using a shallow bed and/or a substoichiometric air supply could control the in-bed temperature sufficiently to limit the extent of limestone calcination in the shale as well as enhance combustion efficiency.

Operational data is not available because the concept has not been incorporated in an operating unit to date. However, METC's preliminary feasibility tests on Israeli oil shale have demonstrated the viability of this concept for oil shales with high limestone content. In addition, the pseudo two-stage combustion appears to be applicable to high-calcium (limestone bearing) oil shales as it offers a means of avoiding the heat-consuming calcination reaction.

FIGURE 10: CONCEPTUAL DESIGN OF AN FBC SYSTEM FOR HIGH-CALCIUM CARBONATE-BEARING OIL SHALE

6.4.3 Shale-Oil Aerosol Recovery

Depending on the shale oil characteristics and the rate of cooling, shale oil aerosols of various sizes are formed. When shale oil is cooled, micron-size aerosols are formed in the condensation process. These aerosols are extremely difficult to collect which represents a potential operational and/or environmental problem. METC has experienced the formation of micron-sized aerosols in coal gasification as well as oil shale retorting processes. Conventional methods of aerosol recovery utilize venturi scrubbers and wet (washed) electrostatic precipitators. These methods are energy consumptive and, in some cases, cause the generation of hard-to-treat wastewater streams. In general, the desired technical features of an oil aerosol collection process are as follows:

- High-collection efficiency which translates into increased oil yield and low levels of hydrocarbon emission into the environment.
- Low power consumption, either directly or indirectly due to, for instance, system pressure drop.
- Minimum water requirements to reduce the capital and operational costs relating to water processing and enhanced environmental and socioeconomic attributes.

METC identified two approaches which potentially provided these features: (1) a membrane filter and (2) a porous metal filter used in conjunction with a glass wool filter. Basically, these processes are effective due to the forced coalescence of tiny aerosols in an "Anderson impactor-like" medium. Unable to follow the flow path of the gas stream, the aerosol collides with other aerosols to form large collectable aerosols. In order to insure sufficient coalescence between the aerosols, the membrane filter and the porous metal filter were selected with the pore size on the order of a few microns.

The membrane filter was selected for use in some of the earlier test runs. Commercially available Teflon® material with an average pore size of about 5 microns was used. The membrane filter surface was tilted at an angle larger than the angle of repose for the shale oil. The purpose was to insure a steady flow of collected shale oil which would, in effect, self-clean the filter surface. During test evaluations, the membrane filter collection system was found to be unsatisfactory and was discarded due to the high frequency of membrane failure. The pressure drop across the membrane filter was relatively high and the membrane filter had a tendency to frequently plug.

In another test series, a porous metal filter of the "bayonet" configuration was adopted for the final stage of aerosol collection. Initial experiments with a configuration having an effective oil recovery showed a pressure drop of 15 mm water column at a superficial velocity of 0.4 cm/sec on the filter surface of the metal filter. The total pressure drop for the system was about 250 mm water column. The configuration for shale oil aerosol collection, using a porous metal filter, is shown in Figure 11. Additional information about the oil collection system start-up and test procedure is presented in Appendix A, Part 4.

Results with the porous metal filter aerosol collection system under the conditions of the retorting experiments are encouraging. Further study is needed to more fully understand the mechanism of aerosol collection and perfect an effective collection configuration.

7.0 CONCLUSIONS

Based on METC's research findings and other published information, fluidized-bed processes were found to offer highly attractive methods to maximize the heat recovery and yield of quality oil from oil shale. The principal reasons are the fluidized-bed's capacity for (1) high in-bed heat transfer rates, (2) large solid throughput, and (3) selectivity in aromatic-hydrocarbon formation.

The METC research program showed that shale-oil yields were affected by the process parameters of retorting temperature, residence time, shale particle size, fluidization gas velocity, and gas composition. (Preferred values of yields, of course, may differ among major oil shales.) The major conclusions are as follows:

- Fluidized-bed combustion/retorting of most oil shales offers a promising method for improved yields and quality.
- The fluidized-bed retorting process enhanced the yield of shale oil rich in aromatic content. The formation of aromatics is highly desirable, preferably because their low hydrogen-to-carbon ratio permits high oil yield in hydrogen-deficient oil shales. Also, aromatic hydrocarbons with anti-knocking properties are desired input components in gasoline fuels and are the building blocks for petrochemical production.

FIGURE 11: POROUS METAL FILTER

- Process parameters can significantly affect oil yield for high-quality hydrocarbon production.
 - Retorting temperature
 - In-bed residence time
 - Shale particle-size distribution
 - Fluidization gas composition and velocity
- Fluidized-bed processing of shale offers high heating rates and a flexibility in choice of retorting conditions which far exceed that available with conventional packed-bed retorting approaches.
- Integration-utilization of the spent shale is the key to the retorting process attractiveness and the twin bed retort/combustion concept is an effective means of combining retorting with combustion.
- For the calcium carbonate-containing oil shales (i.e., Colorado, Israeli, and Moroccan) the sulfur emissions were considerably below the EPA's SO₂ emission limit without limestone addition. For eastern oil shales, Tennessee Celina and Kentucky Sunbury oil shales, with high-sulfur content and low-limestone content, the addition of limestone to insure compliance with the EPA's SO₂ emission is necessary.
- Direct combustion of oil shales is sometimes the preferred utilization approach and combustion of various shale qualities was successfully demonstrated in a fluidized-bed combustor. High-combustion efficiency, 95 percent or better, is attainable for all the tested low-grade and/or eastern shales. The types of shales for which combustion might be preferred are those with low Fischer assay and low sulfur (or high sulfur but also high calcium) content.
- Oil shale has a considerable amount of nitrogen bearing organic compounds. The combustion of the nitrogen bearing compound is likely to form nitrogen oxides to a level in excess of the EPA standards. The reduction of nitrogen oxides can be achieved by the addition or control of carbon inventory in the hot fluidized bed. Staged combustion is also an effective means of control which can be readily incorporated in the design of a fluidized-bed combustion system using oil shales.

- The relatively high fraction of inert materials in oil shales requires the feeding and removing of a large quantity of solid material in both retorting and combustion processes. Specific means for handling the large quantities of solids must be considered in designing a commercial unit. In addition, the spent-bed material contains a considerable amount of the heat extracted from the system and recovery of this heat is usually desired to achieve attractive overall process efficiencies.

8.0 SELECTED LIST OF PATENTS FILED

- 8.1 "Reverse Flow Fluidized-Bed Combustion Apparatus," Jer-Yu Shang, et al.,
DOE Case S-58-521.

Summary of the invention:

A cyclone for recycling solid particulate material located in a combustion zone, the subject combustion apparatus will permit the height of the freeboard to be significantly less than in known fluidized-bed combustors since the combustible fines contained in the freeboard volume are recycled back into the combustion zone to assure that combustion of the combustible material can be essentially fully burned before the spent-particulate material is discharged from the combustion zone. Also, by recycling the particulate matter, the residence time and high freeboard temperatures can be maintained at sufficient values for considerably enhancing the combustion efficiency as well as controlling the emission of environmental pollutants.

- 8.2 "Oil Yields Improvement from Oil-Shale Retorting," Jer-Yu Shang, et al.,
DOE Docket No. S-58-563.

Summary of the invention:

Generally speaking, the retorting process is a process of rapidly heating up the oil shale to cause the thermal cracking of organics. The prompt diffusion of the retorting product vapors outside the limestone-based shale matrix could reduce the extent of secondary thermal cracking of retorting vapor products.

The invention proposes the use of carbon dioxide as a heat carrying fluidizing gas in retorting the

limestone-based oil shales. The chemistry of limestone calcination is depicted by the following chemical reaction:

Carbon dioxide gas acts as a heat-supplying fluidizing medium to the fluidized-bed retorting of oil shales, opens passages throughout the limestone-based and magnesium carbonate-containing oil shale matrix to avoid the undesirable secondary cracking, and is a recoverable carrier gas in oil shale retorting which can be readily removed from the retorting gaseous product stream by a number of commercially available means.

8.3 "Reflecting Fluidized-Bed Combustion," Jer-Yu Shang, DOE Docket No. S-58-597.

Summary of the invention:

This invention proposes the use of a granular-bed filter to retain a layer of glowing fly ash or chars elutriated from the fluidized bed on top of a layer of limestone granulars. The limestone granulars are fed into a slanted screen at an angle no more than the angle of repose. Periodic vibrations and air pulsations will help to spread the limestone granulars in a thin layer evenly on the screen.

The deposition of elutriated glowing flyash and char fines will:

- Form a layer of red hot glowing surface which radiates heat into the freeboard to keep the freeboard temperature sufficiently high for the combustion of combustibles.
- Calcinized the limestone under a high carbon dioxide partial pressure to form large-pored lime for high calcium utilization. Consequently, low calcium-to-sulfur ratio may be achieved for high sulfur retention.

- Reduce the nitrogen oxides, mostly nitric oxide, into nitrogen and carbon dioxide for low nitrogen oxides emission. The glowing or hot flyash and/or char fines in a packed bed of limestone granulars will readily reduce to nitrogen oxide.
- Remove particles (flyash and char fines) by the particle filtration process and, henceforth, diminish the dust loadings in the flue gas which may help to reduce the amount of fabric filter area required or the capital investment of other dust-removing equipment.
- Burn off the unburned condensable hydrocarbons to an extent which may reduce the likelihood of blinding the downstream and fouling of heat recovery systems fabric filter.

8.4 "Loop-Bed Combustion Apparatus," Jer-Yu Shang, et al., DOE Case S-58-582.

Summary of the invention:

The primary objective of the invention is to provide a combustion system utilizing a novel combustor in the form of a continuous loop so that the combustible material introduced into the system is continuously circulated in a dilute phase until the combustible material is effectively consumed. The loop configuration combines the functions of mechanical grinding and combustion. Raw material need be ground only fine enough for the feeding system to function reliably as the interparticle attrition process will reduce sizes which provide fresh surfaces for sulfur sorption and improved combustion. The loop exit is a port on the inward facing surface of the loop which lets the loop itself serve as the first stage of particle/gas separation. In addition, the loop lends itself to both load following and pressurized operation due to inherent modularity (individual loops are either "on" or "off" in a system consisting of multiple loops) and strength characteristics associated with "small" diameters.

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APPENDIX A: TEST PROCEDURES

Several tests are usually conducted during experimental runs to assure normal operation of the test equipment. These tests include:

- Mutual flue gas and oil leakage test between two compartments.
- Controls of operating temperatures, pressures, and particles residence times in both compartments.
- Capacity test for dust removal system.
- Test for efficiency of oil collection system.

A. FLUE GAS AND OIL LEAKAGE TEST

Oxygen in combustion flue gas supports combustion; therefore, it is very important to assure there is no appreciable gas leakage from the combustion compartment to the retort compartment because oil produced from retorting would be oxidized and result in a lower oil yield. To test if there is flue gas leakage occurring, one can monitor the oxygen concentration in the exhausted retorting gas stream. This test can be conducted during the preheating stage of the run while there is only spent oil shale fluidizing in the beds. When gas streams are properly separated, i.e., when leakage of flue gas does not occur, oxygen concentration measured in the retorting gas stream must be higher than the concentration in the combustion gas stream. The difference under test conditions should be approximately 1,500 ppm.

On the other hand, it is undesirable to have oil vapor leakage from the retorting to the combustion compartment. Such leakage can be detected by monitoring the hydrocarbon content in the exhausted combustion flue gas stream during the preheating stage. If it is detected, one should reduce the propane fed to the combustion compartment to maintain a hydrocarbon content in flue gas lower than approximately 1,000 ppm. Although such action will not stop any oil leakage from the retort, trading off some product oil for propane to maintain a normal combustion operation seems economically acceptable. An alternative approach involving stripping of flue gas and oil at the corresponding openings between compartments by using inert gas such as N_2 or steam may be incorporated into the modification of test equipment.

B. CONTROLS OF OPERATING TEMPERATURES, PRESSURES, AND PARTICLE RESIDENCE TIMES

- *Temperature Control* — Temperature control is critical in both combustion and retorting compartments because unstable temperature, as well as non-uniform temperature distribution throughout each compartment, will result in the formation of clinkers. Thus, fluidization in twin-bed type units will be suppressed. Thermocouples are, therefore, installed along each compartment to monitor temperatures of gas in the plenum chamber, above the distribution plate, and along the bed and freeboard regions. In retorting and combustion of 20 x 45 mesh Colorado oil shale, N_2 is preheated to 500°F before it is fed into the bed at the rate of 240 scfh. Air and propane, premixed at a volume ratio of 25 to 1, is fed at a rate of 286 scfh without preheating. At these flow rates of N_2 and the air/propane mixture, temperatures of retorting and combustion compartments can be maintained fairly stable at 900°F and 1,600°F $\pm$ 10°F, respectively. After these temperatures are reached and become stable, raw oil shale is fed, and temperatures of retorting and combustion compartments can be controlled by adjusting operating variables such as fluidizing-gas flow rates, air/propane ratio, or oil-shale feeding rate.
- *Pressure Control* — Pressures in both compartments are usually around the atmospheric pressure in normal operation. To assure stability, a differential pressure gauge should be installed to monitor the pressure difference between two compartments as well as the absolute pressure in either one of the compartments. It is also a good practice to check the exhausted lines of the bed after each experimental run to avoid any plugging problem occurring in the next run. In addition, spent shale must be removed from the combustion compartment through the overflow port on a regular basis during the run to prevent any accumulation of solids in the bed and pressure build-up in the bed. The expanded bed height

of both compartments should be adjusted to the same level of the overflow port so that steady-state operation can be attained shortly after the raw shale is fed to the bed. An important condition of operation necessary for avoiding contamination of retorting products by combustion products is to maintain a pressure equilibrium between the two compartments.

- *Residence Time Control* — In a twin-bed combustor-retorting unit, it is more important to control the particle residence time in the retort side than in the combustor side. This is because longer particle residence time in the retort would result in a greater extent of reaction, i.e., more oil yield for a given amount of shale. On the other hand, the more vigorous fluidization occurring in the combustor provides a very high heating rate of spent shale and makes the particle residence time in this compartment less controlling. In order to increase particle residence time in the present test system, provisions for adjusting fluidization velocity as well as shale feeding rate have been provided. Considering that secondary cracking would occur for too long a residence time, provision for adjustment of the freeboard height and arrangement of different types of nozzles along the solid circulation path between two compartments should be considered as another means of increasing particle residence time in the bed.

C. CAPACITY TEST FOR DUST REMOVAL SYSTEM

It has been determined that the glass wool filter (GWF-I) is a critical step in the system. This filter removes almost 95 percent of all the fines passing through the system. In a system using two cyclones, the GWF-I has been found to be more effective. All the fines leading into this system must be retained and nearly none of the fines carried into the oil collection system. This is accomplished by an adequate amount of glass wool that is closely packed into the filter body in such a way that no by-pass channels between glass wool and the metal walls of the filter are provided. However, glass wool must not be packed too tight or it will create such a high resistance to flow that the vacuum pump cannot be effective. Experience has suggested that some glass wool should be placed at the exit pipe of the filter to detect if there are fines escaping through the filter as the filter approaches its saturation point.

D. TEST FOR OIL COLLECTION SYSTEM

A very crude way to determine if there is any oil vapor escaping from the system to the exhausted vent is to place a piece of white paper at the vent for 30 seconds and watch for oil spots on the paper. No oil spots should be observed after the porous metal filter has been installed.

It has been found that among the three oil-collecting units (i.e., water condenser, glass wool filter [GWF-II], and porous metal filter) the one collecting most of the oil is the porous metal filter. The water condenser primarily condenses the oil from vapor into liquid mist, and a few droplets may coalesce and be collected as usable oil. Following the condenser, a glass wool filter forces the tiny oil droplets in the vapor stream close enough to increase coalescence.

Pressure gauges were installed in the lines upstream and downstream of each filter and pressure drop used to indicate filter saturation. As each filter was saturated with oil, oil in usable form (i.e., bulk liquid) was collected in the collector underneath the filter. Those oil droplets escaping from GWF-II were retained and coalesced on the surface of the porous metal filter. Oil-liquid stream then flowed down to the bottom of the filter for collection in a bottle.

A vacuum gauge installed between the porous metal filter and the vacuum pump provided an indication of the saturation point of the filter.

Before an experimental run, pressure drops across GWF-II and porous metal filters must be checked to make sure that reasonable gas flow is being drawn through the dust removal and oil collection systems.

APPENDIX B: 2-INCH ELECTRICALLY HEATED FLUIDIZED-BED COMBUSTOR

A. EXPERIMENTAL FACILITY

A 2-inch diameter electrically heated fluidized-bed, oil-shale retorting unit (EHFBR) was developed to study the effects of several parameters on percentage yield and composition of the product shale oil. These parameters include retorting temperature, fluidization velocity, particle residence time, particle-size distribution, concentration of CO₂, type of oil shale, and type of oil recovery system. A schematic flow diagram of this test unit is illustrated in Figure B-1. This experimental system is primarily composed of three parts: oil-shale retorting, dust removal, and shale-oil recovery system.

The EHFBR is made of 2-inch diameter, 21-inch long, Schedule 160, Type 316 stainless steel pipe. The expanded bed height is 5 inches. The exterior of the EHFBR is wrapped with an electrical heating element which is capable of maintaining the retorting temperature up to 1,200°F. The intent of externally heating the vessel is to thermally simulate a large reactor (even though the fluid dynamics do not) by achieving lateral isothermality.

The freeboard height of the EHFBR is 16 inches. This is favorable to decreasing the entrainment of fines. The outside surface around the freeboard section is also wrapped with an electrical heating element in order to eliminate cold wall effects.

The distributor is made of a perforated plate with an open area of 1.8 percent. There are 37 holes, each of 0.03-inch diameter, arranged in a triangular pitch of 0.26 inch, and counter-sunk to control the plugging and weeping. The distributor pressure drop is designed at 0.3-0.4 inches of water to assure flow stability and to prevent uneven distribution of gas flow.

The expanded bed height is set at 5 inches by the provision of an overflow drain made of 0.75-inch diameter tube. This drain tube points downward to form an angle of 26 degrees with the vertical axis of the EHFBR. During the operation of this bed, spent shale overflows into a collector through the drain tube. The overflow opening is installed at a pressure balance point where the local pressure is equal to atmospheric pressure.

Two pieces of square trusses are used as internal baffles in the bed to control bubble growth and prevent the bed from slugging. The clearance between the first truss and the distributor plate is 2 inches, as is the clearance between the two trusses.

Oil shale, crushed to the desired size, is continuously fed from an open hopper into the retort by a 0.375-inch diameter screw feeder. The screw feeder is driven by a variable-speed motor. The feeding rate can be controlled to change the solid residence time in the bed.

To get a smooth distribution of fluidizing medium, a plenum chamber with a vertical height of 4 inches is used in the EHFBR. Nitrogen and/or CO₂, which acts as a chemically inert fluidizing medium, is preheated to the desired temperature and is introduced to the plenum chamber below the distributor plate.

It is critical to choose efficient dust separators in order to remove all the dust particles from the oil shale retorting product gases. At the exit of the freeboard, two cyclone separators in series, followed by a glass wool filter, are used to separate the dust from the effluent stream. To maintain the effluent stream temperature, the dust removal system is wrapped with heating tapes.

The key technical problem in the oil-recovery system is to design a system that will result in high oil yield and small pressure drop. After a series of tests, the recovery system for oil mist (see Figure B-1), was developed. An air-cooled condenser collects the heavy shale oil. A subsequent glass wool filter forces the aerosol droplets to coalesce, and larger liquid droplets collect on the glass wool filter. Following the glass wool filter, a porous metal filter with a pore size of 3 microns was installed. Tiny oil droplets, smaller than pore size, pass through the glass wool filter and are filtered and condensed at the inner wall of the case of the porous metal filter. The uncondensable vapors and fluidizing gas are then passed through a low efficiency vacuum pump before being vented into an exhaust hood. The oil recovery system is also wrapped with heat tapes. The temperature of the recovery system can be adjusted according to the oil shale properties and thermal operation conditions to prevent water vapor from condensing and contaminating the product oil.

FIGURE B-1: 2-INCH ELECTRICALLY HEATED FLUIDIZED-BED RETORTING SYSTEM EXPERIMENTAL CONFIGURATION

B. TEST PROCEDURES

- *Fluidization Velocity Control* — The minimum fluidization velocity depends on the relevant physical and chemical properties of fluidizing gas and oil-shale particle size and retorting temperature. It is essential to measure the minimum fluidization velocity of each type of oil shale at different particle sizes.

In our experiments, 1.5 to 2.0 times the minimum fluidization velocity was used in order to improve fluidization quality and to reduce entrainment of fines. The minimum fluidization velocities of different kinds of oil shale are tabulated in Table B-1.

- *Temperature Control* — Retorting temperature is one of the most important factors affecting the thermolysis rate of oil shale and secondary cracking of the oil vapor. To get the desired retorting temperature, the preheated nitrogen temperature and the bed temperatures is controlled by adjusting the powerstats of electrical heating elements in the preheater and on the reactor. Three thermocouples installed in the expanded bed indicated that the temperature gradient in the axial direction was less than 20° to 40°F. It is more important to control the bed temperatures at the very beginning of each run because raw oil shale entering the bed will decrease the bed temperature rapidly.

Temperatures of the gas stream in the freeboard, two cyclones, and the glass wool filter were maintained at 800°F to 700°F by powerstats and heat tapes, according to oil shale properties and operational conditions, to eliminate secondary cracking of oil vapor and plugging of the system by the mixture of condensed heavy oil and dust.

TABLE B-1. MINIMUM FLUIDIZATION VELOCITIES

OIL SHALE	PARTICLE SIZE MESH	BED TEMPERATURE °F	U _{mf} cm/sec
Israeli Oil Shale	30 x 45	800	10.4
Israeli Oil Shale	30 x 45	900	10.5
Israeli Oil Shale	30 x 45	1,000	10.3
Israeli Oil Shale	20 x 30	900	18.2
Israeli Oil Shale	14 x 20	900	30.1
Indiana Oil Shale	30 x 45	800	12.4
Indiana Oil Shale	30 x 45	900	12.6
Indiana Oil Shale	30 x 45	1,000	12.3
Cleveland Oil Shale	30 x 45	900	13.3
Colorado Oil Shale	30 x 45	900	16.5
Michigan Oil Shale	30 x 45	800	10.0
Michigan Oil Shale	30 x 45	900	10.1
Michigan Oil Shale	30 x 45	1,000	10.2
Michigan Oil Shale	30 x 45	1,100	10.4
Sunbury Oil Shale	30 x 45	800	19.3
Sunbury Oil Shale	30 x 45	900	19.2
Sunbury Oil Shale	30 x 45	1,000	19.4
Sunbury Oil Shale	30 x 45	1,100	18.6

It is undesirable to have the water vapor in the retorted gas phase condense and contaminate the shale oil. The temperature of the oil-recovery system was adjusted at 100° to 150°F, according to oil shale properties and thermal operation conditions, to prevent water from condensing.

- *Pressure Adjustment* — A slight change of pressure from atmospheric has insignificant effect on the retorting operation, but the change of pressure in-bed will make it difficult to operate the screw feeder smoothly. Usually a vacuum pump is adjusted to keep the pressure balance point at the overflow where the local pressure is equal to atmospheric pressure. It is more important to regulate the location of the balance point at the very beginning of each run because the pressure drop of the entire retorting system will increase rapidly with the increase of bed material and the concentration of oil mist in the gas stream. The pressure drop of the retorting system will rapidly increase from about 17 to 30 inches of water within about 25 minutes. When the entire system reaches a stable condition (in about 1 hour), its pressure drop, depending on the properties of oil-shale particles and oil mist, will rise to between 40 and 60 inches of water and hold constant.
- *Control of Solid In-Bed Residence Time of Particles* — The mean solid in-bed residence time of particles is one of the most important factors affecting oil yield. In general, the mean residence time of particles depends on the bed temperature, fluidization velocity, and feeding rate. The mean residence time of oil-shale particles in the bed can be calculated by the following equation:

$$t. (\text{min}) = \frac{W}{F} \times 60$$

Where F is the overflow rate of the spent shale which is dependent on the raw shale feed rate (gm/hr) and W is the solid inventory in the bed which is dependent on the fluidization velocity and the retorting temperature (gm/hr). When the retorting temperature and fluidization velocity are maintained at constant values, the mean residence time can be changed by varying the feeding rate.

APPENDIX C: 6-INCH DIAMETER FLUIDIZED-BED COMBUSTOR

A. EXPERIMENTAL FACILITY

The experimental facility and major dimensions of the 6-inch diameter laboratory-scale fluidized-bed combustor are shown schematically in Figure C-1. The laboratory-scale fluidized-bed combustor is a 6-inch diameter, refractory-lined cylindrical vessel. The bed region is 16 inches high. The expanded freeboard section, 8 inches in diameter, is 48 inches high. The 6-inch unit has a conical air-distributor plate, which has an open area of 2.77 percent of the total distributor area. Fluidizing air from the plenum passes through 64 0.125-inch diameter nozzles arranged in three concentric circles. Fuel from the hopper is fed directly into the bed by a 1-inch, variable-speed metering screw, which is cooled by a water jacket.

Limestone, when it is needed, is fed directly into the top of the expanded bed through a 0.75-inch screw feeder. Spent-bed material can be extracted from the combustor through a 0.75-inch drain pipe at the bottom of the conical distributor or removed through the overflow tube that penetrates the side wall at an upward angle of about 30 degrees. The opening of the overflow tube is set at the 4.5-inch level. The overflow tube discharges into a lockhopper which is periodically emptied. During testing, an overflow tube is used to control the expanded bed height. Flue gases are continuously sampled and analyzed after passing through primary and secondary cyclones and being filtered. Flue-gas monitoring equipment includes the instruments tabulated in Table C-1.

TABLE C-1 FLUE-GAS MONITORING INSTRUMENTATION

COMPOUND	PRINCIPLE OF OPERATION	INSTRUMENT
CO	NDIR*	MSA — LIRA Model 303
CO	NDIR*	MSA — LIRA Model 303
SO ₂ NO-NO _x	Flame Photometric Chemiluminescence	Meloy — FPD Thermoelectron Series 10
O ₂	Thermomagnetic	Leeds and Northrup Model 7803 G
Total HC	Flame Ionization	Beckman Model 400

*Nondispersive infrared

B. START-UP PROCEDURE

Start-up of the laboratory-scale unit is accomplished by preheating the lower part of the vessel to the desired temperatures (approximately 1,500°F) with premixed propane gas and air fed through the conical distributor plate into the bed region.

When the vessel reaches the desired temperature, bed material is slowly added to the bed region through the limestone screw feeder. Once the feeding of bed material is completed, fuel feed is started. As the fuel feed is continued, the propane gas is gradually cut off and combustion becomes self-sustaining. The entire start-up procedure for the laboratory-scale unit takes about 1.5 hours.

FIGURE C-1: 6-INCH LABORATORY-SCALE FLUIDIZED-BED COMBUSTOR

APPENDIX D: 9-INCH FLUIDIZED TWIN-BED FOR RETORTING AND COMBUSTION

A. EXPERIMENTAL FACILITY

A 9-inch diameter fluidized twin-bed (FTB) unit was designed for oil shale retorting and combustion tests. A schematic process diagram is shown in Figure D-1. Basically, the entire process can be divided into three systems. They are twin-bed reactor system, dust and oil collection system, and peripheral measurement devices.

- *Twin-Bed Reactor System* — A stainless steel cylindrical reactor with 9-inch internal diameter (ID) is composed of three major parts: plenum chamber, distribution plate, and bed and freeboard regions. The entire exterior of the reactor, including top and bottom, is covered with a cast refractory insulation material of 1-inch thick mass leaving a reactor inside diameter of 7 inches.

The plenum chamber is divided into two chambers: one for N_2 and the other for an air and propane mixture. The volume ratio between these chambers is 1.2 to 1 (N_2 chamber/air chamber). A partition plate between chambers is designed so that N_2 is evenly distributed throughout the entire cross section of the retorting compartment as well as the regions in the combustion compartment near the openings between two compartments. The N_2 curtain is provided at the openings in order to reduce the leakage of O_2 and oil vapor between two compartments. N_2 is preheated by a 3.4 kW heater before it reaches the chamber. Air and propane are premixed in a volume ratio of 25 to 1 and introduced into the combustion chamber without any preheating.

A flat distribution plate has a 3-inch diameter inverted conical pole located at the center surrounded by a total number of 204 nozzles. Each nozzle has a 1/16-inch round opening slanted at an angle of 30 degrees with the plate in order to improve the solid circulation between two approximately equal size compartments. These nozzles are arranged in a triangular pitch. There are 105 nozzles for the retorting compartment and 99 nozzles for the combustion compartment.

The total height of the bed and freeboard regions is 28 inches. Retorting and combustion compartments are separated by a partition plate made of stainless steel. The plate is supported by the inverted conical pole erected from the center of the distribution plate. The pole is installed to provide an annular passage for solid circulation. Its inverted, tapered shape is designed for enlarging the freeboard cross sectional area. At the bottom of the partition plate on both sides of the pole, there is a 3-inch by 0.625-inch rectangular opening to allow bed materials circulating from retorting to combustion compartment through one of the two openings and back to the retorting compartment through the other. The interior of these regions is covered with 2-inch thick firebrick to reduce the heat loss through the reactor wall.

Raw oil shale is introduced into the retorting compartment through the feeding port located near the opening where solid particles are entering from the combustion compartment. Raw shale is fed by means of 0.375-inch screw feeder from a 6-inch hopper. The maximum feeding rate of 20 x 45 mesh Colorado shale using this feeder is 165 g/min. An overflow port is located on the wall of the combustion compartment at a level 10 inches above the distribution plate. The overflow port at this level sets the expanded bed height for materials in the combustion compartment. A sight glass port is also located in the combustion compartment for the use of observing the ongoing combustion inside the bed.

- *Dust and Oil Collection System* — The retorted gas stream exhausted from the retorting compartment contains both solid fines and product oil mist carried by the flue gas. Solid fines must be removed prior to the collection of product shale oil. Due to the large flow exhausted from the retorting compartment, only a portion of flow is split from the main exhaust line and transported through the collection system under the action of a vacuum pump. Normally, approximately 15 to 20 percent of the exhaust gas volume is fed into the collection system.

Two 2-inch ID cyclones in series followed by a 6-inch ID GWF-I are used for removing solid fines from the gas stream. These three units are covered by heating tapes and insulation material (fiberglass) to maintain high temperature in the gas stream passing through this system. The glass wool filter serves

FIGURE D-1: FLOW SHEET OF COMBINED RETORTING AND COMBUSTION OF OIL SHALE IN A FLUIDIZED TWIN BED

as a safeguard to prevent contamination and plugging of any subsequent units. This filter has successfully retained almost all the fines from the exhausted gas stream provided that new glass wool, in the proper amount and correctly installed, is used for every run.

The oil collection system utilized for this 9-inch twin-bed unit is identical to the 2-inch unit described in Appendix B. The startup and operating procedure is also the same.

B. PERIPHERAL MEASUREMENT DEVICES

- A manometer is used to measure the pressure drop between the combustion and retorting compartment.
- Pressure gauges are installed at the inlet and outlet of GWF-II to show if the glass wool filter is plugged by fines or is saturated with oil or if both conditions exist.
- Pressure gauges installed at the gas inlet port to each chamber indicate the pressure drop across the distribution plate and the bed in each compartment in order to evaluate the minimum fluidization velocity at high temperature.
- Thermocouples are installed along the wall of each compartment as well as the plenum chamber. Thermocouples are also installed at the retort exhaust vent, between the cyclone and GWF-I, between GWF-I and the condenser, and at the inlet to the vacuum pump.
- Gas flow through the dust and oil collection system is monitored by a gas flow installed after the vacuum pump.
- A large gas-analysis system has been hooked up to the exhaust gas lines leading from either compartment to monitor the concentrations of CO, CO₂, SO₂, O₂, and HC in these lines during the test run.

APPENDIX E: 18-INCH DIAMETER FLUIDIZED-BED COMBUSTOR

A. EXPERIMENTAL FACILITY

The 18-inch atmospheric pressure fluidized-bed combustor is depicted schematically in Figure E-1. It is basically a refractory-lined cylindrical combustor having an internal diameter of 18 inches in the bed region, which is 45 inches high, and an expanded freeboard section 24 inches in diameter, which is 27 inches high. The combustor is equipped with horizontal-water or steam-cooled heat exchangers submerged in the bed, comprised of six hairpin configurations, 0.5-inch Schedule 160, Type 316 stainless steel tubes. Each cooling loop is fed separately from a water or steam manifold, and any number (from one to six) of these tubes can be in service. Each tube has a surface area of 0.6 square feet. These tubes are used to obtain heat-transfer coefficient data in the bed and, additionally, to control bed temperatures. Because various test objectives may require that some of them be out of service for extended periods of time, they must be physically able to withstand bed temperatures; 160 type stainless steel alloy tubes are thus required.

The freeboard area is equipped with a single-pass heat exchanger with the tubes essentially horizontal in a zig-zag pattern around the periphery of the freeboard space.

Fuel is metered by a 1.875-inch variable-speed screw from a hopper that is suspended from a weigh cell. The fuel falls into a fuel-injection chamber where it is entrained by compressed air at ambient temperature and transported through a 1-inch Schedule 40 pipe through the base of the conical distributor to the nozzle. Velocity through the nozzle ranges from 60 to 80 ft/sec. Feed rates of 280 pph were used with the oil shales. Limestone sorbent, when needed, is fed via a 1.875-inch variable-speed metering screw from a weigh hopper directly onto the top of the expanded bed.

Fluidizing air comes from a plant air supply through a pressure-regulating valve and a panel-operated control valve to the air plenum located beneath the conical distributor. From the plenum it passes through 95 holes 0.1875 inch in diameter which are arranged in seven equally spaced concentric circles into the directional nozzles of the air distributor. The nozzles are 0.125-inch stainless steel 90-degree elbows, each equipped with a drilled pipe plug on the discharge side. These elbows are welded onto the distributor plate and turned at various angles, but all are pointed generally toward the center of the cone. The pipe plugs are drilled to 0.125 inch on the first four circles radially outward from the cone apex, and the plugs in the outer three circles are drilled to 0.0625 inch. Open area amounts to 0.27 percent of the distributor plate area. At 4.9 ft/sec fluid velocity, maximum pressure drops through the distributor and the expanded bed amounts to 3 psig.

Gases from the fluidized-bed combustors are continuously analyzed so that operations can be followed closely. Any potential upset of the bed can be detected and corrected or monitored during the course of a test. The gas analyzers consist of infrared, paramagnetic, chemiluminescent, flame photometric, and fluorescent instrumentation. The outputs from the analyzers as well as other data points are recorded on magnetic tape for use in process calculations. A CRT is available to the operators to be used in communicating with the process computer. This allows retrieval of data and displays plots as needed to monitor the process.

Bed depths vary from 1 to 3.5 feet and are controlled by an overflow tube and lockhopper arrangement, or by a screw, removing material from the bottom of the bed. Bed-depth indicators are based on differential pressure measurements or by use of newly developed capacitance probe. Material removed from the bed is carefully weighed and added to the output for material-balance calculations.

Because of the versatility of the test unit (FBC 18-1) and its capability to operate over a wide range of conditions, it has proven to be a valuable research tool at METC.

B. START-UP PROCEDURES

Start-up of the unit is accomplished by preheating the lower part of the vessel to 1,400°F with premixed natural gas and air fed through the fluidizing-air nozzle into an initial charge of spent bed material. When the 1,400°F temperature level is reached, about 40 pounds of bed material is added at a slow rate until it is also up to temperature. When this occurs, fuel feed is started; the natural gas is shut off as the fire becomes self-sustaining.

FIGURE E-1: 18-INCH FLUIDIZED-BED COMBUSTOR

Feeding of bed material is continued until the desired bed level is attained (usually about 36 inches). The entire procedure from cold start takes from 3 to 5 hours. Bed temperatures are maintained by three methods:

- Bed level which is maintained by the level of the opening in the overflow tube.
- Fuel feed rate which is controlled by the screw-feeder rpm.
- Heat exchangers in the bed.

Bed temperatures can be closely maintained with these controls. Superficial velocities have ranged from 2 to 4.9 ft/sec using 0.125-inch by 0-inch fuel and 0.5-inch by 0-inch fuel. This is controlled by measuring the ambient air-input readings. Sorbent material (such as limestone) is fed directly into the top of the bed by a controllable-speed screw feeder. The pressure within the combustor is equivalent to the few inches of water-pressure drop through the dust-collecting system.

APPENDIX F: GLOSSARY OF ABBREVIATIONS AND ACRONYMS

DOE	U.S. Department of Energy
EHFBR	Electrically Heated Fluidized-Bed Retorting Unit
EPA	Environmental Protection Agency
GWF	Glass Wool Filter
ID	Internal Diameter
METC	Morgantown Energy Technology Center
PONA	Paraffinic, Olefinic, Naphthenic, and Aromatic
TDH	Transported Disengaging Height
TFBRC	Twin Fluid-Bed Retort/Combustor