

# Integration of Large Scale Retorting Operations with Laboratory Testing and Analyses

**Mark T. Atwood, PhD**

Chemist, Consultant  
Westminster, Colorado

## Abstract

Commercial oil shale development is very expensive, but there have been cases where extensive development testing has been done on a large scale without sufficient, preliminary, small scale laboratory and bench scale testing. In addition, when large scale operations are being done, laboratory analyses and testing can be closely integrated with pilot plant and semi-commercial operations to reduce their extent and duration and to provide needed chemical information.

Tosco Corporation conducted retorting testing from the smallest laboratory scale up to pilot plant and 1,000 ton per day operations. Parallel laboratory feed testing, using the Total (Tosco) Material Balance Fischer Assay (TMBA), was done to compare product yields and properties obtained on large scale operations with yields and product properties obtained on a 100 gram scale. Since all the products from TMBA retorting were contained and measured it was possible to obtain balances for organic and inorganic carbon, nitrogen, sulfur and enthalpy and compare them with balances obtained from larger scale operations. An organic carbon balance was particularly useful in following the fate of the kerogen. TMBA testing and product analyses provided a useful baseline for comparisons.

Retorting processes will be discussed where heat is variously supplied by externally heated solids, internal combustion, heated gases and by a variety of in situ means. Yields and chemical and physical properties of products will be compared. The use of laboratory testing and analyses for process guidance will be reviewed.

## Introduction and Purpose

It is the purpose of this publication to place more emphasis on the application of laboratory analytical procedures to assist in the commercial development of oil shale processing. Analytical procedures may be carried out at the process site or may be applied at a more distant central laboratory. The consideration of continuously operating on-line analytical instruments is recommended where ever feasible.

Above ground and in situ processes are reviewed so that analytical applications may be more clearly visualized.

The chemical and physical nature of oil shale is reviewed briefly to assist in designing analytical support options and to explain the basis for calculation of oil shale reserves.

It is fortunate that the kerogen in oil shale is composed mainly of aliphatic structures which are amenable to evolution of hydrocarbon vapors upon pyrolysis. Aromatic structures in kerogen tend to form coke when pyrolyzed. If kerogen were primarily aromatic in nature little oil would be produced upon pyrolysis, and there would be no hope for an oil shale industry.

A major part of this presentation will be a review of laboratory procedures including a review of diagnostic laboratory tools which can be applied to gas, vapor and liquid products to support operating personnel in making process decisions.

## Chemical and Physical Properties of Oil Shale

Oil shale, in the commercially interesting areas of the Green River Formation; covering Colorado, Wyoming and Utah; is composed primarily (ca 85 %) of marlstone and saline minerals bound in a continuous phase of a three dimensional organic polymeric phase (ca 15 %) called kerogen. The purpose of a commercial processing plant is to recover as much of the organic material as liquid oil and gas as possible. This always requires thermal processing.

### *Kerogen*

A typical composition of Green River Formation oil shale kerogen is shown in Table 1 (Smith, 1961). The weight percent kerogen in Colorado oil shale can be calculated by dividing the organic carbon value by 0.805.

Kerogen is usually considered to be a very high molecular weight, three dimensional polymer accounting for its insolubility in common organic solvents, such as methylene chloride,. However, upon treating oil shale at a supercritical temperature (400 degrees C) for one hour with methanol and water, Sam Dorrance at Western Research Institute (Dorrance, 1985) showed that the kerogen could be broken down chemically into a complex mixture which was soluble in common solvents. One test yielded 130 % of Fischer Assay by dissolving the decomposed kerogen in cyclohexane. The author believes that a sizeable portion of the kerogen is locked into the inorganic matrix as carboxylic acid salts and that this factor ac-

Table 1: Composition of oil shale kerogen

<i>Element</i>	<i>wt. %</i>
Carbon	80.5
Hydrogen	10.3
Nitrogen	2.4
Sulfur	1.0
Oxygen	5.8
Total	100.0

counts for the insolubility in organic solvents. This is a significant finding. See below for further discussion.

The chemical composition of kerogen has been shown to be remarkably uniform across the Green River Formation (Smith, 1961). This should be expected of a uniform sedimentary deposit and is significant in reserve calculations over wide areas.

The mineral composition, shown in Table 2, is also relatively uniform with some variations.

Table 2: Mineral composition of oil shale (from Smith, 1978)

<i>Mineral</i>	<i>Weight %</i>
Carbonates	48
Feldspars	21
Quartz	15
Clays	13
Analcime + Pyrite	3
Total	100

The presence of any nahcolite or dawsonite, which decompose endothermally below the usual retorting temperature of 500° C. would be undesirable in retorting operations. Nahcolite and dawsonite are usually not present in significant quantities in Mahogany zone oil shales.

In retorting oil shale it is important that contained calcite and dolomite do not thermally decompose significantly in the range 1050-1150° F. Carbonate decomposition is endothermic, using up process heat, and puts CO<sub>2</sub> into the product gas with a lessening of heating value. Laboratory testing of spent shale to detect a decrease in mineral carbonate or gas analysis showing a higher than expected CO<sub>2</sub> content should be done on site and on a routine basis.

### *Other Important Physical and Chemical Properties*

The best discussion of the subject of physical properties can be found in comprehensive fashion in The Cameron Engineers

compendium, (Cameron Engineers, 1978, pp.24-51).

When dealing with Green River Formation oil shales the calculation of Fischer Assay yield in gallons per ton can be accomplished using the following formula (Smith, 1961):

$$Y = 31.563X(E2) - 205.998X + 326.624$$

where Y= oil yield in gallons per ton

X= specific gravity of oil shale at 60/60(F)

There is also a published relationship between organic carbon content (not total carbon) and Fischer Assay oil yield (Atwood, 1979):

$$Y = (X - 0.913) / 0.442$$

where Y= oil yield in gallons per ton

X= weight % organic carbon in whole oil shale

The above two relationships can be used on Green River Formation oil shales to eliminate the necessity of setting up cumbersome Fischer Assay equipment.

### **Reserve Estimates and Fischer Assay**

Over 20% of the United States is underlain by some form of kerogen containing oil shale. The bulk of this oil shale is located in Colorado, Utah and Wyoming. Most of the development work done in the last 50 years has involved the rich oil shale formations of this tri-state area. These oil shales were formed in sedimentary formations deposited from thin annual accretions of algae. The uniformity of these formations allows lateral extrapolations of oil shale properties over wide areas and limits the number of assays that must be done for resource estimations.

Dozens of cores have been drilled by the U.S. Bureau of Mines, and core segments, usually at one - foot intervals, have been prepared and subjected to Fischer Assay to give potential yields of oil in gallons per ton. This exercise resulted in the accumulation of a mass of data which was the basis

for reserve calculations used in various Federal leasing programs.

#### *Fischer Assay*

Fischer Assay, where oil shale is retorted under standard conditions, produces product gas, liquid oil, water and leaves spent shale as the residual by-product. In doing the hundreds of assays for reserve estimates only the oil product is collected, weighed and, with determination of specific gravity, calculated as gallons per ton of oil shale. This procedure has been described in several U.S. Bureau of Mines publications: Stanfield, R.I. 3977 (1946); Stanfield, R.I. 4477(1949); and Hubbard, R.I. 6676 (1965).

For these procedures to give meaningful data care must be taken with sample preparation of the 100 grams of oil shale used for assay. The above referenced Reports of Investigation describe techniques for sample preparation.

The results of all of this extensive assaying work leads to the conclusion (Cameron Engineers, 1978 pp.3,4) that the Green River oil shale formation of Colorado, Utah and Wyoming covers approximately 16,500 square miles and contains a total of roughly four trillion barrels of potential shale oil.

#### *Tosco Corporation Methods*

Tosco Corporation refined the Fischer Assay procedure further by using a stainless steel retort into which an aluminum can, containing the 100 gram oil shale sample, was inserted. Preparation of representative oil shale samples requires extensive grinding, blending and sieving (Goodfellow, 1974).

The retort was electrically heated, and the temperature of the oil shale was controlled using a thermocouple which was placed in a tube welded to the side of the retort. Feedback from the thermocouple was used to keep electrically produced heat adjusted to the prescribed time-temperature pattern (Goodfellow, 1974). All retort products, except gas, were collected, measured and assayed. The product gas was vented into a hood, since toxic hydrogen sulfide was part of the product mixture.

In order to assist Tosco II pilot plant and semi-works operations, the Tosco Material Balance assay (TMBA) apparatus was developed. (See the preceding reference.) In this procedure all of the retort products were collected and measured. Product gas was collected from the retort through a solenoid valve into an evacuated glass container. Product gas was analyzed by gas chromatography. Material balances were routinely obtained, and, after standard analyses, balances were calculated for organic carbon, mineral carbon, nitrogen, sulfur and higher heating value. Similar assays and analyses were done for pilot plant and semi works operations using the same oil shale source. The close similarity of assays and balances obtained from large-scale semi-works (1,000 tons/day) and laboratory TMBA operations is illustrated in the preceding reference. (See Table 3) Close balances and assay comparisons were routinely obtained. In addition, we routinely obtained near 100 % closures on organic carbon, sulfur and nitrogen balances. The balance comparisons between the TMBA products and the 1,000 ton per day semi-works products matched each other closely. (Goodfellow, 1978).

In order to get a first approximation of commercial Tosco II yields and product analyses at a selected Piceance Basin site, coring, core sample preparation and TMBA can be carried out. This procedure would eliminate the initial need for expensive pilot plant operations.

It is interesting that the results obtained in

Table 3: Products of the Tosco II System – 1,000 ton/day semi-works (Lbs. Yield/100 Lbs Fischer Assay Oil)

Products	Semi-Works	TMBA
C3 & lighter	10.8	11.7
C4 & heavier	105.8	104.1
<i>Subtotal</i>	<i>116.6</i>	<i>115.8</i>
Acid Gases	9.9	10.3
<i>Total</i>	<i>126.5</i>	<i>126.1</i>

Also good organic carbon, sulfur and nitrogen balance comparisons were routinely obtained.

the Fischer assay *batch* retorting procedure closely match those obtained from large-scale, *continuous*\_Tosco II operations. Having the TMBA results provided a valuable check on pilot plant and semi-works results. Significant differences between the two could be due to poor sample preparation, the presence of extraneous materials such as water, sand and nahcolite, and pilot plant or semi- works malfunctions.

Fischer Assay and TMBA testing would be valuable as a reference point when processes other than Tosco II are being run. This recommendation would apply to *in-situ* as well as surface processes. It is important that great care be taken with sample preparation so that representative samples are assayed.

### Advanced Modes of Surface Retorting

Surface retorting always involves two activities which are expensive and subject to environmental problems. These are oil shale mining and spent shale disposal. The bulk of the spent shale may exceed the volume of the mined oil shale.

The five processes which have been extensively investigated are listed in Table 4.

The **Tosco II** process (Whitcombe, 1982, pp.153-160) and Cameron Engineers, 1978, pp.85-88) was investigated intermittently over 25 years. A 24 ton/day pilot plant, located near Denver, was used to bring the process to a near commercial stage. Final process experience was gained in a 1,000 ton/day semi-works plant located on Parachute Creek near Grand Valley. The author was involved as laboratory manager in close support of both facilities.

Significant improvements have been made to the process since closure of the semi-works. These were made using the 24 ton per day Tosco II pilot plant and other pilot plants located in Golden, Colorado. Ex-Tosco engineers and chemists are available for assistance in commercialization. The technology is currently owned by Conoco-Phillips, and technical help is available from

Table 4: Advanced modes of surface retorting

Name	Retorting Vessel	Heating Medium	Largest Scale
Tosco II	Rotary Kiln	½ inch Ceramic Balls	1,000 Tons/Day
Unocal B	Vertical Retort (Bottom fed)	Hot Recycle Gas	Goal 11,000 Tons/Day
Lurgi	Screw Mixer*	Burned Spent Shale	One ton/day (Colorado shale)
Paraho	Vertical Retort	Direct or Indirect	1.1 Ton/hour
Petrosix (Brazil)	Vertical Retort	Hot Recycle Gas	Commercial (260 metric tons/hr)

\* Tested on five Colorado oil shale types.

their engineering staff in Bartlesville, Oklahoma.

The **Union B** process (Barnet, 1982, pp. 169-182) used hot recycle gas for process heat. This replaced the earlier combustion unit designed to produce process heat by burning retort feed and products. Both systems were bottom fed by a rock pump. The Union B process was operated near Grand Valley at a scale approaching commercial for a number of years and was closed down in 1991.

Both Tosco and Union activities involved extensive, large - scale room and pillar mining and crushing. This important technology is available for future application. (See the Tell Ertl collection at the Colorado School of Mines library.)

The **Lurgi Process** for oil shale is based on many years of experience in thermal processing of coal (Weiss, 1982). In the Lurgi Ruhrgas process for oil shale, preheated oil shale, at about 300-400° F, is mixed with 1200-1300° F shale ash in a horizontal screw mixer., Retorting is carried out in a few seconds at 900-1000° F (482-537° C). Vaporized shale oil is condensed. Product gas can be used for fuel or for the production of hydrogen. The Lurgi Ruhrgas Process has been tested on five Colorado oil shale feeds with no apparent problems.

The **Paraho Process** (Lukens, 1989) has been under continuous development for decades. The pilot plant at Anvil Points has been used intermittently during that time. The process involves a vertical kiln with oil shale flowing vertically through

a preheating zone, a retorting zone and a combustion zone. A version involving indirect heat by hot recycle gas has been tested extensively (ibid). Pilot plant capacity is normally about one ton per hour. At this writing the pilot plant is in an active phase.

The **Petrosix Process** (Hohmann, 1982) is in commercial operation in Brazil using local Irati oil shale as feedstock. The process operates in a vertical kiln using hot recycle gas to provide process heat. . The Petrosix process was tested on Colorado oil shale on a small scale. In 1991 the Brazilian plant was producing 3,000 bbl/day of oil. Stone and Webster Engineering of Denver (Ed Piper) was formerly the United States representative. Much of their files are available in the Tell Ertl Collection at The Colorado School of Mines library.

### Advanced In Situ Retorting Processes

Problems of oil shale mining and crushing and spent shale disposal are eliminated or minimized when in situ processes are used. Various in situ processes have been tested and evaluated since the earliest days of oil shale development. Some of the major processes being investigated currently are listed in Table 5.

The **Geokinetics** horizontal process was thoroughly tested in 26 retorts. These were located near the surface. Process heat was provided by burning rubblized oil shale in a horizontal fashion. A total of approximately

Table 5: Advanced *in-situ* processes

Name	Direction of Flow	Heat Source	Scale of Production
Geokinetics	Horizontal	Burning rubbleized oil shale	26 field tests, 40,000 bbl. of oil
IITRI	Vertical (downward to sump)	Radio Frequency	Field tests in Utah (oil product mostly naphtha, kerosene and gas oil)
Shell	Horizontal	Electric	See 26 <sup>th</sup> Oil Shale Symposium
EGL	Vertical	Hot liquids	See 26 <sup>th</sup> Oil Shale Symposium
VMIS	Vertical to bottom sump	Combustion of rubbleized shale	10 and 150 tons at LETC large field tests by Occidental

40,000 barrels of oil were produced during the extended operations (Lekas, 1979).

The **IITRI process (Illinois Institute of Technology Research Institute)** uses radio frequency heating (Sresty, 1982). The electric power required can be generated miles away to mitigate air pollution at the process site. Field and laboratory tests show that oil can be produced at a low temperature of 385° C if enough time is allowed. This oil is rich in light components, such as naphtha and diesel. For example (Gould, 1984) described oil produced at 385° C with a high API gravity of 34.4 (specific gravity of 0.85) and a C5+ naphtha content of 15 volume %, a kerosene content of 29 % and a gas oil content of 26 %. The API gravity is high, and the low boiling point fraction distillation cuts are also large, indicating a much lighter product than that which would be produced from surface retorting. Low sulfur and nitrogen contents in the IITRI product oil point to increased ease of refining.

Product oil, as produced, could be tested on site by gas chromatographic simulated distillation analysis to detect any changes in boiling range and thus product value.

The **Shell process**, designated the Mahogany Research Project, was discussed at this 26<sup>th</sup> Oil Shale Symposium by Harold Vinegar. It involves underground electric heating. Extensive field tests are currently underway.

During field operations product gas samples may be taken and subjected to the diagnostic testing described below. Product oil samples may be quickly and routinely

evaluated using boiling point column gas chromatography. The expected low boiling point character of the mixture can be easily confirmed.

The reader is referred to the Shell web site, Shell.com. Extensive long term field and engineering tests are currently underway. See also Shell U.S. Patent 6,991,032 (January, 2006).

The **EGL process** involves passing hot heat transfer liquids, such as Dowtherm and Syltherm, from the surface through heat transfer pipes placed vertically through the oil shale formation. Produced oil and gas are collected at the bottom of the formation and brought to the surface for separation of the heat transfer liquid and further processing of the hydrocarbon liquids and gas. The reader is referred to the web site, [www.eoilshale.com](http://www.eoilshale.com).

The technology was discussed at this 26<sup>th</sup> Oil Shale Symposium by Paul Lerwick.

The **VMIS (Vertical Modified In Situ) process** has been studied over a number of years by the Laramie Energy Technology Center (LETC) and by Occidental (Chang Yul Cha, 1982). The rubbleized combustion zone is directly above the retorting zone, and hot combustion gases flow from top to bottom. The LETC experimented with 10 and 150 ton simulated above ground retorts, achieving 76 and 66 percent yields based on approximated Fischer assay.

The Occidental VMIS field tests involved some mining to create void space and blasting to produce rubble. Large scale tests were run on a property near DeBeque,

Colorado. One test run produced 30,000 barrels of oil. During operations oil and gas samples can be removed often for laboratory diagnostic testing, using procedures described below.

## Diagnostic Measurements for Controlling Process Performance

During in situ operations it is difficult to sample and analyze for produced oil and spent shale because of the irregular release of oil product and the difficulty of sampling for spent shale. However, the product gas by necessity issues regularly and can be easily sampled and analyzed as produced. Much information on retort performance may be gained from careful measurement and analysis of the issuing gas stream. Produced oil may issue continuously or intermittently from the process and may be easily sampled.

Diagnostic concepts for oil and gas may be divided into two main categories:

### 1. Ethylene to ethane ratios

Jacobson, *et al.*, at the Laramie Energy Technology Center reported that, in oil shale retorting, the products produced depend on both the temperature range used and the residence time in the reaction zone. This was based on laboratory tests on Colorado, Utah and Wyoming oil shales. A Retorting Index was derived:

$$T = \text{Retorting Index} = 1,000/[0.8868 - 0.4007 \log (\text{ethylene/ethane})]$$

The units of Retorting Index are in degrees Fahrenheit. The ethylene to ethane ratio (weight percent) increases as retorting temperature and residence time increase. A quick review of this equation shows that as the ratio of ethylene to ethane increases the Retorting Index will increase.

This diagnostic tool would be applicable to both surface and *in situ* retorts. Results by on-line gas chromatograph to measure the ratio of weight percent ethylene to ethane should give quick indications of any retort temperature changes and changes in residence time.

Results also show that there is a linear relationship between increased Retorting Index and the cracking of hydrocarbon vapors. Cracking results when the retort temperature is raised and the residence time in the retorting zone increases.

### 2. Ratios of higher molecular weight 1-alkenes to n-alkanes and ratios of naphthalene to alkane hydrocarbons

With the use of gas chromatography, information may be gained on variations in retort temperature, residence time, and the effects of coking, cracking and combustion.

When the pyrolysis temperature exceeds the optimum level a lower yield of oil can be expected due to cracking, resulting in (Burnham, A.K. and J.E. Clarkson, 1980) increases in the following ratios:

- ethylene/ethane
- propylene/propane
- toluene/C7 and C8 paraffinic hydrocarbons
- p-xylene/C8 and C9 paraffinic hydrocarbons
- naphthalene/ C11 and C12 paraffinic hydrocarbons

Coking can be reduced by adding a purge gas to diminish residence time. Steam is particularly effective.

The oil yield, exclusive of loss due to coking, cracking and burning (burning would only apply if a combustion retort were being used), can be expressed as:

$$\text{OYL} = (1-f_c) \times (1-f_b) \times 100\%$$

The term,  $f_c$ , is the fraction equivalent to Fischer Assay oil yield which is destroyed by coking. The term,  $f_b$ , is the fraction of oil vapor which is destroyed by combustion and cracking.

The amount of oil vapor which is cracked, coked or burned can be estimated by examining the ratio of naphthalene to C11 and C12 straight chain hydrocarbons which become n- hydrocarbons and were previously attached to the naphthalene structure as alkyl groups. Alkyl substituted naphthalenes are thermally less stable than the aromatic naphthalene nucleus present.

These alkanes produced during operations can be determined analytically by use of capillary gas chromatography. This instrument should be located and operated on site to assist in plant operations.

An estimation of the fraction of oil coked may be estimated (Burnham, A.K and J.E. Clarkson, 1980) by measuring the ratio of 1-dodecene to n-dodecane. High ratios indicate low coking yields. As the fraction of oil coked increases, the ratio of 1-dodecene to n-dodecane decreases. It is also feasible to measure coke yield by scraping coke from the reaction vessel and weighing it.

### Free Process

The author would like to suggest that personnel working in the field of oil shale development take advantage of the work reported by Sam Dorrance (1985) of The Western Research Institute. This process used a methanol/ water mixture at high pressure to literally extract the kerogen from the inorganic matrix to produce an organic solvent soluble kerogen.

The process steps envisioned are as follows:

1. Treat oil shale at 400 degrees C for one hour with a methanol/water mixture under pressure at supercritical conditions.
2. The kerogen becomes 90 % liquid (130% Of Fischer Assay).
3. Add methylene chloride to dissolve the solubilized kerogen. (Methylene chloride is an excellent organic solvent with a boiling point of only 40 degrees C. Other organic solvents may be selected.)
4. Settle or centrifuge to remove the inorganic residue.
5. Distill off the methylene chloride and recycle it back to the process.
6. Send the heavy, remaining liquid product to a coker. This is done with Canadian oil sands processing.

7. Refine the coker distillates to produce salable petroleum products.
8. Use residual product coke as process fuel.

Companies or groups looking for an entry into the oil shale processing field should consider this process.

### Summary

We have reviewed the important chemical and physical properties of oil shale and have noted factors, such as the high aliphatic character of the kerogen which allow pyrolysis to produce significant yields of liquid oil, vapor and gas. The major above ground and in situ processes which have been investigated extensively have been reviewed.

The procedures which were used to estimate potential oil reserves from the tri-state Green River Basin were outlined. The Green River formation of Colorado, Wyoming and Utah contain over four trillion potential barrels of shale oil.

The TOSCO II process and its development through pilot plant and 1,000 ton /day semi-works illustrated the value of close laboratory support using Tosco Material Balance Assay for yield and product comparisons which matched the large scale operations.

The direct application of on site analytical capabilities to diagnose conditions in the various operating retorting processes have been discussed. These diagnostic tools permit rapid feedback from laboratory testing to operations personnel which should shorten expensive development time.

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