METHOD OF LABORATORY ANALYSIS FOR OIL SHALE ASSAY

by

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I. INTRODUCTION

Oil shale is much commoner than is generally realized, occuring on every continent and in every geological system. They are misnamed, but it is unlikely that any more appropriate name will be acceptable either to geologists. Few important deposits actually consist of shales and even fewer have yielded any oil. In the conventional sense most "oil shale" is actually bituminous, nonmarine limestones or marlstones containing kerogen.

Only a few marine examples can be properly described as shales. The common composition involves about 50 percent of mineral carbonates by weight. A variety of silicates may also be present, possibly derived from the reaction of circulating alkaline waters with volcanic debris.

A. *Background*

A forecast of the world energy demand and supply, whether or not the energy hungry world scenario occurs later rather than sooner, will depend on the policy approaches adopted by the oil and gas industry as well as oil prices, discoveries of sources, energy conservation and alternative energy sources.

Initially, some of the main concerns facing the oil and gas industry in the future are:

- The immediate shortage of oil (energy hungry world).
- The foreseeble decline in oil reserves.
- The need to find alternative liquid fuels.
- Synthetic crude that can be produced by retorting oil-shale, is a potential supplement to crude oil.

B. *Hypothesis of Oil-Shale*

Fisher (2) investigated that the pyrolysis products composition produced from oil-shale heating of Green River. The result is shown in Tabel 1.

In proposing this study three types of hypothesis considerations are:

a. Experimental: (Scientific Australia, April 1980)
   - In general oil-shale contains C (carbon) constituents,
   - Shale as source rock having rich C content,
   - Pyrolysis of oil shales above 932° F yields kerogen (a heavy oil),
   - Oil shale is in plentiful supply in many parts ofthe world.

b. Empirical
   - World oil reserves (proven + probable) = 700 billion bbls,
   - World residual oil reserves: 300 billion bbls,
   - World shale-oil reserves: 30.000 billion bbls,
   - Worldshale-oilproducers e.g. USA, Brazil, ex USSR, Canada, Italy, China, France, Germany, Thailand, UK, Luxembourg, Estonia, Australia,
   - Australia produced >50 liters/ton (Queensland-Rundle formation).

c. Implemental:
   - 1973 : In Estonia (Kukersite Shale), produced 10.000 bbls/day from 26 million tons of shales,
   - 1910-1934 : In Tasmania (Tasmanite Shale), produced 10.000 bbls shale-oil from 41.500 tons of shales,
   - 1977 : In Brasil (Rico Blanco projected modified in situ (M.I.S) retorting):
     - 57.000 bbls/day from underground retort
     - 19.000 bbls/day from surface retort.
   - 1972 : In Colorado (after 17 years research) using M.I.S (modified in situ) retort:
     - 60.000 bbls/day from underground retort
     - 55.000 bbls/day from surface retort.

Table 1
Pyrolysis product composition of oil-shale from green river formation, Colorado

Product constituents	Weight % of organic shale compositions
Oil	63
Gas	15
Carbon residual	13
Water vapour	9
Total =	100.0

II. PROCEDURES FOR OIL SHALE ASSAY

Laboratory standard operation procedure (Pyrolisis method), for producing oil content producing from oil-shale is by preheating at 932° F to yield the heavy oil (Kerogen). In world commercialy this kerogen, through the refinery procces can produce gasoline, diesel oil and jet fuels. The application of oil shale laboratory technique is best performed in the following steps:

1. Sample Preparation

A. Select a representative piece of the interval (usually one foot) to be assayed. It is highly desirable to use a complete vertical section of a one-foot interval. If the samples submitted do not allow such procedure to be used, select pieces from various vertical positions in the sample interval.

B. Crush the sample in a chipmunk crusher with the jaws set as closed together as possible.

C. Fraction the total crushed material with the sample splitter until a fraction of the crushed material that weighs about 125 grams is obtained.

D. Pulverize the sample so that the material will just pass an 8-mesh screen.

E. Air dry the pulverized sample. After pulverizing, place the sample in a shallow pan and allow to dry for 24 hours to essentially constant weight (it is not necessary to take several repetitive weights).

2. Oil and Water Distillation Analytical Procedures

A. Clean retort cups the welded-on condensorsand the internal cups. Put 2 asbestos discs in the caps and cover with a copper disc.

B. Loosely assemble an empty retort cup with condensing stem, an empty inner retort cup, and prepare cap. Weigh underneath the large Mettler balance to nearest 0.1 gram, then record in column 17.

C. Tare out the inner retort cup on the large Mettler balance. Add 100.0 grams of pulverized, Air-dried sample. Enter grams of sample used in column 20.

D. Carefully lower the inner cup with the weighed sample into the retort cup. Place the retort cup in the special vise provided and screw on the cap very tightly.

E. Position the assembled retort cup, lid and sample in a retort which is at room temperature.

F. Assemble two 50 ml receiving tubes together with two hole rubber stopper and a short stainless steel connecting "U" tube. The short end of the tube should be inserted into one stopper (with the end even with the bottom of the stopper) to be placed in the receiving tube which will be placed under the condensing stem of retort.

The other end of the connecting tube should be inserted into the other stopper so that the two receiving tubes will be submerged to the same depth in the condensing bath.

G. Tightly wrap about one gram of glass wool around and near the end of the "U" tube in the second tube and fluff it under the stopper to cover the second stopper hole real well.

H. Obtain the tare weight of the receiving tube assembly on the balance. Weigh to the nearest 0.01 gram. Record the weight in Column 2.

I. Position the receiving tube assembly on the condensing stem so that about one inch of the condensing stem is below the stopper.

J. Raise the condensing bath so that the water level is about half-away up the rubber stopper. Maintain the condensing bath at -3° C (26.6° F)

K. Turn on the retort. Allow the temperature to rise so that a temperature of 750° F is attained in about 15 minutes. Continue retorting at 750^o F for an additional 15 minutes. Raise temperature of retort to 940^o F and continue retorting for 50 minutes.

L. Lower the condensing bath. Allow the receiving tubes to hang on the condensers for an hour (if time permits). A propane torch flame may be used to heat up the condensing tubes to facilitate drainage.

M. Remove the receiving tube assemble, dry the water from the outside of the tubes and weigh on the balance to the nearest 0.01 gram. Record the weight

in column 1.

N. Cetrifuge the receiving tubes. Usually only the one under the condensing stem will need to be centrifuged, as it is the only one with oil and water.The second tube will normally contain oil only.

O. Read the volume of water to the nearest 0.1 ml. Usually all the water will be in receiving tube under the condensing stem. Record the water volume in column 4.

3. Specific Gravity Analytical Procedures

A. Place the receiving tube from under the condenser in the hot water bath at 100° F.

B. Weigh a 1.0 ml pipette which has been preheated to 100^o F on the balance. Weigh to 0.002 gram. Record the wight in the collumn 11 of the Data Sheet. (As an alternative, you may prefer to tare out the empty pipette on the balance.)

C. Fill the pipette with oil from the centrifuge tube in the water bath to the calibration mark on the neck. Use the manual pipette filler provided so that the pipette is filled precisely to the reference mark,and is not over filled to cause hang up ofoil on the pipette walls.

D. Weigh the filled pipette on the balance to the nearest 0.002 gram. Record the weight in column 10 of the Data Sheet. (If you choose the alternate technique of taring uot the pipette weight on the balance, record the weight of the full pipette directly in column 12 as the specific gravity of the oil at 100° F).

4. Spent Shale, Gas Plus Loss, and degree of coking Analytical Procedures

A. Weigh the cooled retort cup with condensing stem and retorted sample on the balance to the nearest 0.1 gram. Record in column 16.

B. Open the retort cup and pour out the remains of the oil shale. Observe the remains for tendency to coke. The Bureau of Mines station in Laramie reportsthat there is essentially no tendency to coke when the oil content is less than 30 gal/ton. They report 4 catagories : none, slight, medium, and high. A set of values for this qualitative test will have to be developed through experience. Record by checking appropriate column on the Data Sheet to the right of column 21.

5. Data Calculation

A. Specific Gravity: (Column 10) - (Column 11)

1. Step(3-D)- Step(3-B),in grams = Specific Gravity of oil at 100° F/60° F.

   This is valid because the pipettes are calibrated with water at a temperature of 100° F and are volumetrically calculated for volume using the absolute density of water at 60° F. The pipettes have been found to have a volume of 1.000 ml ± 0.002 ml at the stated reference conditions.Calculate to nearest 0.002. Record in column 12.

2. Add 0.015 to the specific gravity at 100° F/60° F to obtain a 60° F/60° F value.

   Record in column 13.

   The Bureau of Mines reports that they find the specific gravity values to vary from 0.88 to 0.96. In the event that an insufficient amount of oil is recovered to measure specific gravity, assume a specific gravity of 0.92.

B. Oil content, gal/ton:

   (Column 6) x (Column 7) : (Column 20)

1. For 100 gram sample charge.

a. Step(2-M)- Step(2-H) -(2-O) = Weight of oil recovered, grams. Record in column 5.

b. Step (5-B1a):Step (5-A2)= Volume of oil recovered, cc. Record in column 6.

c. Step(5-B1b) x 2.397 = gal/ton.Calculate to nearest 0.1 gal/ton. (This istrue because the gal/ton conversion factor divided by 100 is equal to 2.397).

2. For other than 100 gram sample charge

$$\frac{Step(4-1b)\ x\ 239.7}{Grams\ of\ sample\ charge} = gal/ton$$

3. Record in column 8.

C. Oil Content, Weight per cent:

   (Column 5) x 100 + (Column 20)

1. For 100-gram sample charge

   The weight of oil recovered, Step(5-B1a)ingrams is equal to the oil content in weight per cent.

2. For other than 100-gram sample charge Step(5-B1a)

$$\frac{Step(5-B1a)}{Grams\ of\ sample\ charge} = Oil content\ in\ weight\ percent$$

3. Record in column 9.

D. Water Content, gal/ton:

   (Column 4) x (Column 7)

1. For 100-gram sample charge

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Step(2-0) x 2.397 = gal/ton. Calculate to nearest 0.1 gallon per ton.

2. For other 100-grams sample charge

$$\frac{Step\,(2-0)\,x\,239.7}{Grams\,of\ sample\ charge} = gal/ton$$

3. Record in column 14.

E. Water content, Weight per cent:

(Column 4) x 100 : (Column 20)

1. For 100-grams sample charge

The weight of water, Step(2-O), in grams is equal to water content in weight per cent.

2. For other 100-gram sample charge

$$\frac{Step\,(2-0)\,x\,100}{Grams\,of\ sample\ charge} = Wate\,content\,in\,weight\,percent$$

3. Record in column 15.

F. Spent Shale, Weight per cent:

(Column 18) x 100 : (Column 20)

1. For 100-gram sample charge:

Step(4-A)- Step(2-B), in gram + Spent shale, weight per cent, Record in column 18.

2. For other tha 100-gram sample charge:

$$\frac{Step\,(4-A)\ x\,Step\,(2-B)}{Grams\,of\ sample\,charge} = x100 = Spent\ shale, weight\ percent$$

3. Record in column 19

G. Gas Plus Loss, Weight per cent:

100 - (Column 19) - (Column 15) - (Column 9)

1. For 100-gram sample charge:

100-Step(5-F1)-Step(5-C1)-Step(5-E1) = Gas Plus Loss in weight per cent

2. For other than 100-gram sample charge:

100-Step(5-F2)-Step(5-C2)-Step(5-E2) = Gas Plus Loss in weight per cent

3. Record in column 21.

Note: All the required arithmetic functions are shown in the column headings on the data sheet.

H. Reporting:

1. Oil content, L/ton
2. Oil content, weight per cent
3. Water content, L/ton
4. Water content, weight per cent
5. Oil specific gravity @ 60°/60°
6. Spent shale, weight per cent
7. Gas plus loss, weight per cent
8. Tendencyto coke - asdescribed by visual inspection.

III. CASE STUDY OF OIL SHALE DETERMINATION

The implementation of laboratory technique for oil shale determination as desdribed above, is performed on selected samples from West Sumatra (S. Junjung Area).

Initially, the oil shale measurements were performed on shale samples which were taken from S. Junjung area (West Sumatra). In this process, the samples were reduced in size to less than 8 MESH sieve screen using a mortal and pestle, then they were placed in a retort oven (see Figure 1). The oil and water extracted from each sample was measured volumetrically, at equilibrium conditions as shown in the Figure 2. The results calculated data were presented in Table 2, and summarized in Table 3.

IV. EVALUATION

If the data is inspected considering all of the samples grouped together in order of oil indication, show irregularities trends of heavy oil recovery. Irregularity around these general trends can be explained on lithological grounds by variations in sorting and the presence or absence of bituminous materials.

Results of oil shale laboratory measurements on surface samples from West Sumatra (Sjunjung Area), indicate an average data as follow:

1. Oil content, L/ton = 50.113
2. Oil content, weight per cent = 4.5116
3. Water content, L/ton = 44.094
4. Water content, weight per cent = 5.760
5. Oil specific gravity @ 60°/60° = 0.8348
6. Spent shale, weight per cent = 80.60
7. Gas plus loss, weight per cent = 10.128
8. Tendencyto coke - asdescribed by visual inspection. = medium.

In general, the results of laboratory measurements indicate that heavy oil recovery from 1.5 L/ton up to 99.2 L/ton and averaging approximately 50.113 L/ton or approximately 13.19% weight (see Table 3).

Table 2
Laboratory data oil shale study

Location: Sjunjung (West Sumatra)

Spl. No.	Tube+rock Wiight (gr)	Tube Weight	Water S SG=1	Water vol. (cc)	Oil wt. (gr)	Oil vol. (cc)	Water wt. (gr)	Oil vol. (gal/ton) (% wt.)	Oil content (grm)	Pipet+oil wight (grm)	Pipet weight (gr/cc)	Oil SG 100/60 60/60	Oil SG Correct. (gal/ton)	Water content (Gal/ton)	Water content (% wt)	Cup+rock wight (grm)	Cup weight (grm)	Spent shale (% wt.)	Sample weight (grm)	Gas plus loss (% wt.)
0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
HA-1	37,885	32,941	1.00	4.80	0.144	0.17	4.80	0.407	0.144	25,593	24,746	0.847	0.862	11,506	4.80	472.4	382.5	89.9	100	5,256
HA-2	43,553	32,941	1.00	4.80	5,512	6.99	5.10	16,755	5,512	25,535	24,746	0.789	0.804	12,225	4.80	471.5	382.5	89.4	100	0.288
HA-3	43,137	32,941	1.00	6.00	4,196	5.64	6.00	13,519	4,196	25,490	24,746	0.744	0.759	14,382	6.00	449.8	382.5	67.3	100	22,604
HA-4	45,886	32,941	1.00	3.60	9,345	10.93	3.60	26,194	9,345	25,601	24,746	0.855	0.870	8,629	3.60	455.7	382.5	73.2	100	13,855
HA-5	41,105	32,941	1.00	4.80	3,364	3.89	4.80	9,324	3,361	25,610	24,746	0.864	0.879	11,506	4.80	465.7	382.5	83.2	100	8,639

Remark:

Column 1 = Input weight data
Column 2 = Input weight data
Column 3 = Water spesific gravity equals 1
Column 4 = Reading Water volume
Column 5 = Column 1 - Column 2 - Colimn 7
Culumn 6 = Column 5 : Column 13
Column 7 = Column 3 x Column 4
Column 8 = Column 6 x 239.7 / 100
Column 9 = Column 5 : 100 x 100%
Column 10 = Input weight data
Column 11 = Input weight data
Column 12 = Column 110 - column 11
Column 13 = Column 13 + 0.015
Column 14 = Column 7 x 239.7 / 100
Column 15 = Column 7 : 100 x 100%
Column 16 = Input weight data
Column 17 = Input weight data
Column 18 = (Column 16 - Colum 17)/100 x 100%
Column 19 = Input weight data
Column 20 = 100 - Colum 18 - Colum 9 - Colum 15

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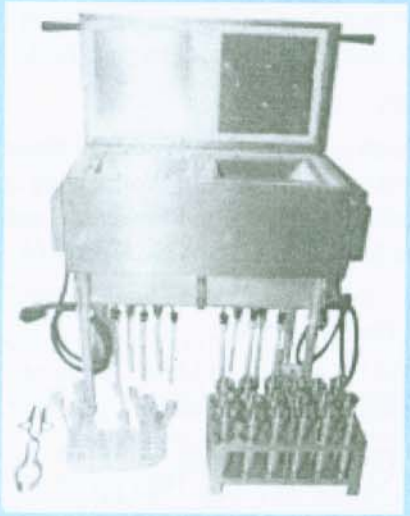

Figure 1
High temperature retort oven unit

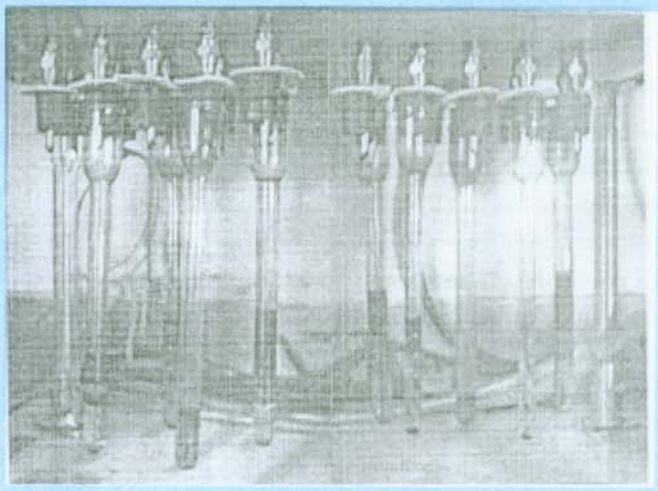

Figure 2
Heating process of oil shale samples using laboratory retort oven
- Kerogen (dark colour)
- Formation water (white colour)

Table 3
Summary oil shale laboratory data

Location: S. Junjung (West Sumatra)

Sample number	oli		Oil specific gravity	Water		Gas plus loss weight %	Spent shale weight %	Tendency to coke
	L/ton	Weight %		L/ton	Weight %			
HA-1	1,540	0.144	0.862	43.55	4.80	5,256	89.9	Slight
HA-2	63,418	16,755	0.804	46,283	4.80	0.288	89.4	High
HA-3	51,169	13,519	0.759	54,436	6.00	22,604	67.3	Medium
HA-4	99,163	26,199	0.870	32,661	3.60	13,855	73.2	High
HA-5	35,291	9,324	0.879	43,550	4.80	8,639	83.2	Medium

Remark:
Degree of "Tendency to coke" : Non, Sli (Slight), Med (Medium), Hi (High)

Reference :
1 gallon (Imperial) = 4.55 literes
1 gallon (US) = 3.785 literes

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V. CONCLUSION

From the above discussion, the following points can be summarized:

1. With the outlook of energy hungry world and for seeble decline in oil reserves as a main source of energy, we have to focus attention on the need to find alternative energy.
2. In some developed countries, the experiment and implementation of oil shale industry, have provided heavy oil from shale to supply additional energy consumptions.
3. This oil shale assay, can be used as a reference to oil shale study in the laboratory and can be developed for further study.
4. Results of oil shale laboratory measurements on surface samples from West Sumatra (Sjunjung Area), indicate oil content is from 1.5 L/ton up to 99.2 L/ton and averaging approximately 50.116 L/ton or averaging approximately 13.19% weight, which can be classified as profitable to produce (see Table 3).

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