

THE FLUIDIZED RETORTING OF OIL SHALE FROM
THE MAHOGANY LEDGE OF THE GREEN RIVER
FORMATION OF COLORADO

by

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I. INTRODUCTION

With the increasing expansion of the uses of easily obtainable concentrated fuels and their gradual exhaustion, future progress as well as national security demand that prospective fuel resources be investigated. Oil shale, oil sand, and coal are the possible materials which can be used to supplement the supply of petroleum.

Shale oil, since its first commercial use, has presented the undesirable aspect of being obtained from a material, oil shale, which contains varying amounts of pyrobitumen, often giving very poor yields for the amount of material processed. It has been found that relative small quantities of the oil yielding material, pyrobitumen, can be extracted by solvents. All true oil shales must be processed by heating to temperatures between 500 °F and 1100 °F. The large amount of material which must be processed to obtain the shale oil, the high sulfur content, and the high boiling fractions found in shale oil provide little impetus for commercial development of the shale oil industry.

Until recently no commercial method had been developed for the continuous retorting of large amounts of oil shale. Therefore, the production of shale oil was hindered considerably. The fluidized solids method seems to possess the greatest possible use.

It was the purpose of this investigation to evaluate the use of the fluidized solids technique for the retorting of oil shale in a 4-inch diameter fluidized bed. Dry gas at approximately 715 °F was used as the fluidizing medium. Mass superficial gas velocities were used as required for the fluidization of oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200-mesh Tyler screen scale.

II. LITERATURE REVIEW

Considerable work has been done on the recovery of oil from oil shale by retorting, however, it was not until 1942 that a continuous retort, applying fluidized solids technique, was introduced into this field of oil recovery. The following review of literature includes a summary of the information on the formation and composition of oil shale, distribution of oil shale, and previous observations of the retortable material in oil shale, kerogen. Also this literature search contains the principles of fluidisation, heat transfer in a fluidized bed, and applications of fluidisation in industry.

Oil Shale

Oil shale is a strong tenacious marlstone⁽²⁷⁾, a sedimentary rock, containing organic matter, which for the most part, is unaffected by solvents but is capable of yielding hydrocarbons on heating. Shales containing this organic matter range in color from a gray to a dark brown, almost black, depending on the proportion of organic matter present. Although there is no outward feel or appearance of oiliness, thin pieces of rich shale can be ignited with a match and will burn with a sooty flame, producing an odor similar to that from petroleum⁽³⁴⁾.

Oil shale has been defined by the American Society for Testing Materials⁽¹⁰⁶⁾ as a compact rock of sedimentary origin, with ash content of more than 33 per cent and containing organic matter that yields oil when destructively distilled, but not an appreciable amount of oil when extracted with ordinary solvents for petroleum.

Formation of Oil Shale. In general, oil shales were formed in open basins in which the deposition of inorganic sediments went on without important or long continued interruption during the accumulation by growth and/or alluviation of the vegetation that formed the carbonaceous and pyrobituminous matter in shale. Because of the large number of oil shale formations within the United States and the world, only the geological formation of the oil shales in the Green river basin are considered because of their present economic possibilities.

History of Oil Shale Formation in the Green River Basin. The Green river formation of Colorado and Utah is a series of lake beds of the middle Eocene age, roughly sixty million years ago, that now occupy two broad, shallow, simple structural basins known as the Piceance Creek Basin in Colorado, and the Uinta Basin in Utah⁽⁶⁾. These two lake basins replace what was at one time one large lake basin, known as Uinta Lake. This lake occupied 10,000 square miles and in this great lake algae flourished and built bedlike reefs

that expanded broadly over the lake floor. Fish, mollusks, crustaceans, and aquatic insect larvae were also plentiful in the lake, and turtles, crocodiles, birds, and small camels, as well as many winged insects, frequented the shores. Along some shores there were marginal swamps, which persisted for some length of time. Thus organic matter of every type was deposited in the lake basin, and due to the strongly reducing medium or ooze on the lake bottom, organic matter was deposited and undisturbed for many years. On top of this organic matter, minerals were being deposited continuously, inclosing the organic matter in a protective layer of sedimentary rock.

Formation of the Retortable Material in Oil Shale.

Many theories (6,17,37,67,74,99), have been set forth as to the alteration which took place upon organic matter to produce coal, petroleum, and kerogen, the retortable material in oil shale. Essentially a bituminisation of plant and animal life resulted in the formation of oil, coal, and kerogen, depending on how completely the process was carried out as to the products received. Kerogen was the result of incomplete bituminisation and is a solid amorphous, organic material which can be distilled⁽³⁴⁾.

Composition of Oil Shale. Oil shales from different parts of the world and in different geological formations differ in both physical and chemical properties. Table I gives analyses of typical oil shales of the United States. Table II tabulates the ultimate analysis of oil shales and related materials from different parts of the world. These tables show the variation in the different oil shale formations.

Kerogen, the Retortable Material in Oil Shale.

Brown⁽⁶⁹⁾ describes kerogen as the material in Scottish oil shale which, on destructive distillation, yields oil. Cunningham-Craig⁽¹⁷⁾ describes kerogen as a material existing as globules which were formed by small drops of petroleum that formed gels with the inorganic matter present. McKee⁽⁶⁹⁾ states that kerogen is neither petroleum nor bitumen, but a substance, pyro-bitumen, yielding petroleum and nitrogenous compounds when it is distilled. He further states that kerogen varies in composition with different oil shales. Thorne⁽¹⁰⁸⁾ describes kerogen as a complex organic mixture, not a definite compound, that the content and composition kerogen varies with different oil shales. Kerogens, however, have the common property of reacting to form a liquid hydrocarbon oil on distillation of the rock. Oil vapors appear at temperatures

TABLE I

Analysis of Oil Shales From Various Localities in the United States

Source of Shale	SiO ₂ wt %	Fe ₂ O ₃ and Al ₂ O ₃ wt %	CaO wt %	MgO wt %	Volatile Matter and Fixed Carbon wt %	Oil Yield gal/ton
DeBeque, Colorado	44.7	25.6	17.65	5.2	40.0	42.7
Elko, Nevada	65.5	25.5	0.60	0.8	33.5	32.5
Dragon, Utah	45.8	16.4	23.90	7.9	42.2	41.6
Green River, Wyoming	38.9	12.4	38.30	4.9	34.9	23.4
Green River, Wyoming	41.9	18.8	17.60	10.9	48.1	58.7
Kentucky	52.0	19.9	12.50	8.2	29.3	18.3
Ione California	43.0	38.1	8.70	2.5	62.9	52.0
Casmalia, California	75.8	13.3	1.40	0.9	18.0

Gavin, M. J. and L. H. Sharp: Physical and Chemical Data Relating to Colorado Oil Shale, Bureau of Mines Report No 2152, p. 3 (1920).

TABLE II

Chemical Analysis of Oil Shales

Shale	Oil Shale Analysis					Ratio	Oil Yield
	C	H	N	S	Ash	C/H	
	wt %	wt %	wt %	wt %	wt %		gal/ton
Scottish (Broxburn)	19.51	2.48	1.36	3.31	72.65	7.86	23.3
Scottish (Pumpherson)	24.88	3.67	0.68	0.80	61.90	6.78	31.0
Australian (Commonwealth)	63.58	7.81	0.81	0.43	22.96	8.14	10.8
Utah (Soldiers Summit)	13.51	1.70	0.39	0.28	66.12	7.94	16.8
Nevada	37.65	5.43	0.39	1.08	46.21	7.24	86.8
Nevada (Elko)	8.61	1.44	0.48	1.12	81.59	5.98	8.4
Colorado (Grand Valley)	23.67	3.50	0.66	1.78	66.60	6.76	63.5
California (Ione)	50.95	5.77	0.42	2.12	22.68	8.83	51.8
Lignite (Black, U.S.)	54.91	6.39	1.20	32.54	8.95
Crude Shale Oil (Autun, France)	79.70	11.80
Crude Petroleum (Heavy Penna.)	84.90	13.70

Note: The following symbols used; C denotes Carbon, H denotes Hydrogen, N denotes Nitrogen, and S denotes Sulphur.

McKee, R. H. and R. T. Goodwin: Organic Matter in Oil Shales, Ind. Eng. Chem., 15, 343-9 (1923).

of 482 °F to 662 °F, and all kerogen is converted if the temperature is increased from 842 °F to 1022 °F.

Action of Solvents on the Kerogen in Oil Shales.

The understanding of the composition of oil shale kerogen would be greatly advanced if any considerable part of it were soluble or preferentially soluble in any given solvent. Little work of this nature has been reported however, and solubility studies have shed little light on the composition of kerogen. McKee⁽⁷⁴⁾ reported that the solubility of kerogen in carbon tetrachloride, acetone, benzol, and chloroform was very low and differed in oil shales of different origin. Gavin and Aydelotte⁽²⁹⁾ ground oil shale samples to pass a 20-mesh screen and extracted these samples with carbon tetrachloride, carbon bisulfide, ethyl alcohol, benzene, acetone, and chloroform. The solvents were then evaporated from the extract and the remaining materials were similar in appearance for the various solvents, being dark brown or black, and more or less fluid. The residues were not appreciably affected by cold concentrated sulfuric acid or nitric acid, and were insoluble in cold ethyl alcohol. The percentages of kerogen extracted varied from traces to 10.98 per cent by weight of shale, and up to 55.8 per cent of the distillation yield. The largest amounts of kerogen were extracted

by acetone and chloroform. Gavin and Aydelotte concluded that the per cent of kerogen extracted by solvents is no criterion of distillation yield.

Action of Chlorine on Kerogen in Oil Shale. Hart⁽⁷⁰⁾ has shown that chlorine, bromine, and iodine are adsorbed by different coals, indicating that in different coals unsaturated organic compounds existed in different proportions. McKee⁽⁷⁵⁾ performed chlorination experiments to determine if unsaturated hydrocarbons existed in oil shales, and found that chlorination of oil shale increased the solubility of hydrocarbons in absolute alcohol from 1.49 per cent, before treating, to 6.23 per cent after treating. Shales used in McKee's experiments were from DeBeque, Colorado, assaying 15.42 per cent shale oil by weight.

Microscopic Study of Oil Shale. Under the microscope all shales show a distinct coloration, which varies in the different bands of shale and in different formations. In thin sections this coloration is usually a reddish-yellow and ranges to a deep reddish-brown in thicker sections and in very rich shales⁽⁷²⁾. The coloration is very selective, as shown by the fact that the grains of quartz and calcite are not affected but appear clear and white, while the argillaceous matrix is deeply impregnated with the coloring matter. The

clay matrix is exceedingly fine and occurs in laminae which are thinner than the laminae of organic material of the section. Under higher magnification these bands resolve themselves into a series of more or less discontinuous thin laminae, of which the brown and yellow or dark laminae are longer and more continuous than the gray or colorless ones. The mineral matter is very finely divided and careful inspection shows that the dark laminae are usually not easily resolved into granules; while the lighter ones are made up chiefly of particles of crystals of mineral matter. McKee⁽⁷⁶⁾, on examination of thin sections of oil shale, found the presence of vegetable debris as films or laminae which extend into the very fine clay matrix. In some of the shales he found that a dark brown structureless material was present and concluded that the material originated in a collection of plant debris which had by decomposition passed into a jelly-like phase, such as found in peat deposits. Thiessen⁽⁹⁸⁾, in a microscopic study of the Devonian oil shales, found the organic matter to consist very largely of spore-exines. Three kinds of spore-exines were reported to be readily recognizable at a magnification of about two hundred diameters, with the most conspicuous being the *Sporangitea Huronensis*, but this type was present only in small numbers. The entire

mass appeared structureless to Thiessen at two hundred diameters, but at much higher magnification the smaller spore-exines were shown very intimately intermixed with fine clay.

Distribution of Oil Shale in the United States. The deposits of oil shale which, by reason of their geographical position and high kerogen content, are of the most immediate promise are those of Colorado, Utah, Wyoming, and California. These deposits do not constitute the total available oil shale in the United States however, and in the following paragraphs the distribution of oil shales within the United States is discussed as to their geographical position in the eastern or western part of the country.

Eastern United States Oil Shale Distribution. The work of Ashley⁽¹⁾ shows that east of the Mississippi river, oil shales exist in very large quantities but are widely distributed. They occur mainly in the Ordovician, Devonian, and Carboniferous systems, from New York to Alabama and westward to the Mississippi river. The most important body of oil shale is the Chattanooga, New Albany, or Ohio shale of the Devonian age. This bed, commonly referred to as New Albany shale, is reported to underlie the eastern coal fields and to crop out in a long line from central Alabama northwestward through Tennessee and Virginia. West

of the Appalachian coal fields its outcrop extends from north to south across central Ohio, through central Kentucky to Louisville, then in a broad belt northward across Indiana nearly to Chicago. From this western belt of outcrop the shale extends eastward under eastern Ohio and underlies nearly all of Kentucky and Indiana west of this outcrop. The average oil yield of samples taken was 10.5 gallons per ton of shale, with some of the samples associated with coal seams as high as 20 gallons per ton.

Western United States Oil Shale Distribution. Of the more prominent shales for their yield of shale oil are those of the Colorado, Utah, and Wyoming. These shales are of the Green river formation, having a total thickness of several thousand feet and an area of more than 1000 square miles⁽⁷³⁾. Recently Belser⁽¹⁰⁸⁾, estimated that an area of 16,500 square miles exists in adjoining portions of Colorado, Utah, and Wyoming and that the portion of this formation assaying more than 15 gallons per ton of shale contains 300 billion barrels of oil. In this formation there are two seams of shale which are considered of economic importance. The primary bed is 500 feet thick and yields an average of 15 gallons of oil per ton, and the lower ledge, known as the Mahogany Ledge, is 70 feet thick and yields

approximately 29.2 gallons of oil per ton of shale. These layers are not the total of the Green river formation which produce oil, but only those considered economical to develop⁽²⁷⁾. For a complete picture of the stratification occurring in the Green river formation see Figure 1, page 15. This formation does not comprise the total of oil shale available in the western United States. The oil shales of California are not considered true oil shales as most of the bituminous matter present can be extracted by suitable solvents⁽⁶⁸⁾. In the southern part of Nebraska there are small deposits of the Cretaceous formation yielding over 40 gallons of oil per ton. These deposits are not of commercial value at present, because their thickness is only a few feet⁽⁶⁷⁾.

Retorting of Oil Shale

The process of heating oil shale to obtain the thermal decomposition and vaporisation of its solid organic matter is called retorting. The organic matter, kerogen, is decomposed at temperatures ranging from 600 to 900 °F, and if heating is continued to a little over 900 °F, all of the kerogen is converted to a mixture of oil vapor, fixed gases, and solid organic residue. Because of the many ways in which oil shale can be

Figure 1

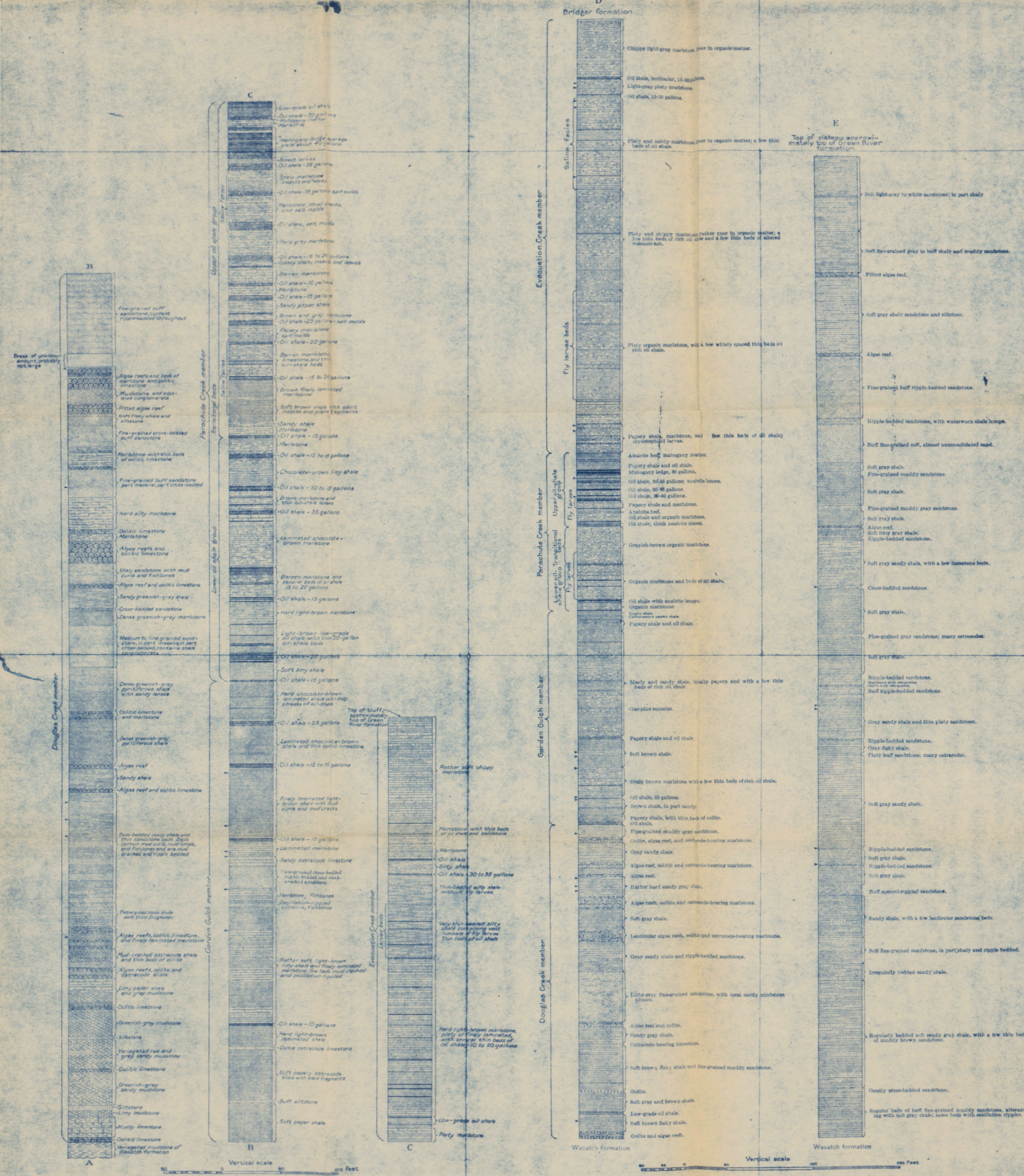
Columnar Section of the Green River Formation

Bradley, W. H.: Origin and Microfossils of Oil Shale of
the Green River Formation of Colorado and Utah,
United States Geological Survey Professional Paper
No 168, (1931).

(see pocket on back cover)

Figure 1

Columnar Section of the Green River Formation



A, B, C, Composite section of the Green River formation. The lower part of the section (the type section of the Douglas Creek member) was measured near the head of Trail Creek, Garfield County, Colo., and the upper part was measured on Cathedral Creek, sec. 26, T. 3 S., R. 99 W., Rio Blanco County, Colo.

D, Composite section of which the Douglas Creek and Garden Gulch members were measured in Hole Hole Canyon, sec. 23, T. 10 S., R. 25 E. Salt Lake meridian, Uintah County, Utah, and the remainder in the canyon of White River, sec. 27, T. 9 S., R. 25 E., Uintah County, Utah.

E, Section of the delta and shore facies of the Green River formation measured a few miles north of Deep Channel Creek in sec. 29 and 30, T. 4 N., R. 95 W., Moffat County, Colo.

heated and handled, many different retorts have been invented, and the patents that pertain to retorting number into the thousands⁽²⁸⁾.

History of Oil Shale Retorting. The history of retorting dates back centuries and the results received on subjecting coal, peat, torbanites, and oil shale to destructive distillation has been exploited by many. France is credited with having developed the first commercial exploitation of oil shale⁽¹⁰⁷⁾. Using a process patented by Selligues, in which superheated steam was used as the source of heat, the French shale oil industry began in 1839. It was not until 1847, however that commercial production of oil, by retorting, was introduced by James Young in England⁽⁷²⁾. Later in 1850 Young's process was introduced into the United States, where, under license, oil shales, cannel, and coal from Virginia, Kentucky, Missouri, and some imported Torbanhill material were retorted. In 1862 the retortable material yielding large amounts of oil became scarce in Scotland, and oil shales were used to carry on the industry. In 1925 over 28 different types of retorts were reported by McKee⁽⁷³⁾ as being in operation within the United States, and during World War II the United States became concerned over the fact that demands for oil had increased tremendously, and knowing of our diminishing reserve capacity to produce oil, enacted legislation that authorized the United States Bureau of Mines to establish

research centers on oil shale retorting as well as other means of liquid fuel production⁽³⁴⁾. In 1942 the Standard Oil Company of New Jersey developed a retort applying the fluidised-solids technique⁽⁴⁾. In 1947 the Bureau of Mines began retorting operations using the Green river oil shale of Colorado. At present there are no known retorting operations being carried out in the United States on a commercial scale.

The Process of Retorting Oil Shale. Retorting in connection with the production of liquid fuels is essentially a low temperature process. One of the most difficult problems of retort design is the design of a heater to heat the material at a sufficiently rapid rate without subjecting the evolving gases to temperatures materially higher than those at which they are evolved. According to Halse⁽³⁷⁾ coals, torbanites, and oil shale are highly nonconductive, and in some of the earlier retorts, heat transfer through the entire bed of shale was not uniform.

Gradual Heating of the Oil Shale in the Retort. If an individual particle of oil shale in a retort is considered it is readily noted that upon heating, whether by contact with the retort surface, neighboring hot material, or by hot gases, the heat will penetrate from the outer surface of the material to the interior. When the outer surface reaches a definite temperature, gases

will be evolved. There will be a definite temperature gradient from the hot exterior of the particle to the cooler interior, and the steepness of this gradient will depend on the rate of heating. The result of such a temperature gradient is that the gases evolved from the interior of the particle will have to pass through a hotter zone than found required to evolve the gases, in order to escape. If the exterior surface of the particle is much hotter than the interior, or the temperature is much higher than that required for retorting, the gases escaping from the interior of the particle will be cracked as they pass through the outer hot region. However, if heating of the material is carried out slowly, the gases evolved come in contact with surfaces not much hotter than the temperature required for evolving the gases. In any case the rate of heating is dependent upon the products desired and the size of particles in the retort⁽³⁸⁾.

Quick Removal of Vapors to Prevent Cracking. The disadvantages of cracking are two-fold, first the volume of permanent gases are increased at the expense of liquid products, reducing them in quantity, and second, the unsaturated hydrocarbons are unstable to some extent. The removal of gases can be accelerated by passing some additional gas into the retort to aid the vapor flow.

According to Halse⁽³⁹⁾, producer gas or steam can be used for this purpose both of which add heat to the retort, and reduce the external heat required for the process. Young first applied the idea of introducing steam into the retort, with the desire to increase the yield of ammonia from the process, and it was later found by Young that the quantity of permanent gases was also reduced by such practice⁽⁷³⁾.

Temperature Control Within the Retort. Effective control of the temperature in the different parts of the retort is of fundamental importance, and has a direct bearing on the yield of oil. Stewart and Trenchard⁽⁹⁶⁾ determined that the greatest yield of oil from oil shale occurred at temperatures below 600 °F, and that the temperature across the retort could be controlled best by applying agitation to the contents. In order to obtain uniform temperature distribution across the retort, Blanding and Roetheli⁽⁵⁾ applied the fluidized-solids technique to retorting, obtaining shale oil of good quality.

The Effect of Oil Shale Plasticity on Retort Operation. The plasticity of oil shales, torbanites, coals, and cannel coals at temperatures below 600 °F denotes a material of high oil content, but the disadvantage of such rich materials is that without some mechanism within

the retort to keep the substance in motion, coking on the walls occur. This coking lowers the heat transfer through the retort wall as well as decreasing the effective surface of the retort. Coking necessitates partial shutdown for cleaning, whether a vertical or a horizontal retort is used. Accompanying this stoppage of operations is a loss of time and heat. Plasticity is caused by the formation of liquid bitumen in the oil shale, and being of a fine colloidal clay, shale is readily decomposed into a suspension of clay particles in liquid bitumen which solidifies on cooling⁽⁴⁰⁾.

Products of Oil Shale Retorting and Shale Oil

Refining. Crude shale oil, as produced from the retorts, resembles crude petroleum as pumped from the ground. Refining of the crude shale oil follows, in general, the methods used in refining other petroleum crudes. The characteristics of shale oil vary considerably with the method of retorting and the raw material used. Crude shale oils may be classified as a paraffin, asphaltic, or mixed base similar to the classification of petroleum crudes⁽³⁾. As examples, the oils from Scotch, Australian, and Manchurian shales are of paraffin base; while those from Estonian, Swedish, and Tasmanian shales are asphaltic. Oils from Colorado shales have a higher percentage of unsaturates than the Scotch and Australian

oils, but less than those from Estonia and Tasmania. Oil from Colorado shale may be classified as a mixed base oil. Guthrie and Schramm⁽³⁵⁾, in retorting oil from Colorado shale, report that crude shale oil is composed mainly of hydrocarbons, consisting of olefins, paraffins, naphthenes, and aromatics, but that appreciable amounts of sulphur, nitrogen, and oxygen compounds are present. Although shale oil bears some resemblance in several of its physical and chemical properties to coal tar, and in others to petroleum, many of its characteristics are so unlike either coal tar or petroleum that it must be placed in a separate category. From the standpoint of refining, Table III, page 22, compares shale oil from Colorado shale with a typical Mid-Continent crude.

Hydrocarbons and Hydrocarbon Products from Shale Oil. Table IV, page 23, shows various percentages of hydrocarbons and hydrocarbon products recovered from shale oil of the Scottish shales. These figures are not however, indicative of the yield of oil products from shale, as the method of retorting has a direct effect on the types of hydrocarbons in the final product. Gasoline obtained directly from shale oil without cracking has an octane rating of about 75, and when hydrogenated the octane rating is increased⁽⁹⁷⁾. Fanning⁽²⁴⁾ reports

TABLE III

Comparison of Mid-Continent Petroleum and Shale Oil from
Bureau of Mines Retort

	Shale Oil	Oklahoma City Crude Petroleum
<u>Crude</u>		
°API at 60 °F.	20.40	39.00
Sulphur, per cent	0.78	0.14
Nitrogen, per cent	1.97
Pour Point, °F.	80	5
Viscosity, SUS at 100 °F.	200	45
<u>Distillation Summary, Per Cent</u>		
Naphtha	3.6	28.4
Light Distillate	19.8	25.0
Heavy Distillate	25.6	18.6
Residuum	49.8	26.3
Loss	1.2	1.7
<u>Naphtha Properties, Per Cent</u>		
Tar Acids	3.0
Tar Bases	9.0
<u>Components</u>		
Paraffins	61
Naphthenes	34	29
Olefins	50
Aromatics	16	10

Guthrie, B. and L. Schramm: Extraction and Refining of Shale Oil, Mech. Eng., 41, 710 (1950).

TABLE IV

Yield of Refined Products from Scottish Oil Shale

Products	Weight Per Cent
Naphthas and Gasoline	9.9
Illuminating and Burning Oil	24.8
Gas and Fuel Oil	24.4
Lubricating Oils	6.6
Paraffin Wax	9.5
Still Coke	2.0
Loss and Gases (includes tar)	22.8
	<hr/>
Total	100.0

McKee, R. H. and others: "Shale Oil", p. 112, The Chemical Catalog Company, Inc., New York, N. Y., (1925). 1 ed.

that by cracking the heavy residuum it is possible to obtain gasoline yields from shale oils ranging from 35 to 55 per cent; while cracking to coke results in 50 to 60 per cent in yields. Higher yields were obtained by hydrogenation of the shale oil before the gasoline was separated from the residuum. These yields were about 80 weight per cent gasoline with an octane number from 65 to 67. He further stated that the lead susceptibilities of these hydrogenated gasolines were high and with the addition of three milliliters of tetraethyl lead per gallon, the gasoline may have octane numbers in the range of 83 to 85. Hydrogenation may also be used to produce diesel fuel, lubricating oil, and paraffin-wax fractions in yields of almost 100 weight per cent of the residuum. Fanning believed hydrogenation to be the most successful method of refining shale oil.

By-Products of Shale Oil Refining. Whatever the source of the organic matter in oil shale, it always produces nitrogenous compounds on destructive distillation⁽⁷³⁾. The actual percentage of nitrogenous compounds produced is small, and varies with the oil shale. Sulphur is found in percentages of about 0.5 of the raw shale. The sulphur and nitrogen compounds are many and vary with the organic matter in the shale. The largest yield of by-products are ammonium sulphate, waxes, tars,

and tar acids. There also exists a number of aliphatic and aromatic alcohols and acids, phenols, sulphur compounds, oxygen and nitrogen compounds, and isoprenoid compounds in oil shale. These compounds, some of which are only found in micro quantities, are the result of kerogen degradation⁽⁹⁹⁾.

Principles of Fluidization

According to Campbell⁽¹³⁾, the practical application of fluidization has so far preceeded its scientific study, since its development and primary industrial application in 1942, that no complete and agreed upon body of laws can be presented. Extensive studies have been carried out, however, with regard to obtaining more information as to the types of fluidized beds, mechanism of fluidization, characteristics of fluidized beds, effect of solid characteristics on fluidization, heat transfer in the fluidized bed, and mathematical relationships involving these variables. The following is a review of literature involving such research.

Definition of Fluidization. Patterson⁽⁹¹⁾ defined fluidization as that unit operation in which a mass of solid particles, usually finely divided, is maintained, by means of an upwardly moving gas stream, in a turbulent dense state. Murphy⁽⁸⁹⁾ went further to separate fluidization

into particulate and aggregative fluidization. He defined particulate fluidization as fluidization where further increases in gas velocities give progressive separation of particles which remain individually and uniformly dispersed, with the bed expanding only to a limited extent; while aggregative fluidization was defined as fluidization in which a portion of gas tends to accumulate in gas pockets, forming aggregates of the solids.

Mechanism of Fluidization. The following is a review of fluidization mechanism with consideration being given to the forces acting in the fluid bed of solid particles, gas flow in a bed of fine solid particles, and the behavior of a bed of solid particles at gas velocities greater or less than minimum fluidization velocity.

Forces Acting in a Fluidized Bed. Patterson⁽⁹¹⁾ stated that within a fluidized bed of solids, the particles are in a continual state of violent agitation that causes them to move in all directions within the confines of the bed, including being projected against the walls of the containing vessel. He further stated that fluidization is produced in a dense bed of solids having a range of particle sizes by collisions between the upwardly moving fine particles and the downwardly moving coarse particles. Murphy⁽⁸⁹⁾ believed that the solid particles were supported by the gas velocity

through the solids bed. According to Wilhelm and Kwauk⁽¹⁰³⁾, fluidization begins when the dispersive forces, the upward viscous drag of the fluid, are balanced by the force tending to retain the bed compact, the downward weight of the particles.

Patterson⁽⁹¹⁾ stated that the friction loss for the gas flowing through the fluidized bed is usually negligible, with the pressure drop due almost entirely to the kinetic energy required to overcome the free fall of the solid particles. Hariu and Molsted⁽⁴²⁾, however, pointed out that friction loss between the solid particles and the container wall was a large factor in pressure drop, but Leva⁽⁴⁸⁾ stated that fluidization occurs at a definite mass velocity, independent of the chamber diameter.

Gas Flow in a Bed of Solid Particles. Ergun and Orning⁽²¹⁾ compared the flow of a gas through a fluidized bed of coke particles with that of the flow of a gas through a randomly packed column of coke of the same particle diameter. They reasoned that if the behavior of the packed bed is followed when a gas stream is introduced from the bottom, the pressure at first increases with the flow rate without bed expansion, or the fractional void remains unchanged. When the flow rate is sufficient to cause a pressure

gradient equal to the buoyant weight of the solids per unit volume, further increase in flow rate causes the bed to expand or to increase in fractional void volume. Up to certain gas flow rate, the rate of expansion does not involve channeling or bubble flow, provided that the column is subjected to some continued vibration. When channeling or bubble flow is not present the bed can be said to be partially fluidized. Until the gas flow is sufficient to cause bubble flow there is very little mixing of solids or particle circulation.

Behavior of a Bed of Solid Particles at Less than Minimum Fluidization Velocity. At less than minimum fluidization velocity Ergun and Orning⁽²²⁾ found, as did Matheson⁽⁶¹⁾, that the gas percolates through the bed without agitation or mixing of the particles, and the pressure drop is less than that equivalent to the weight of bed. Leva⁽⁵⁰⁾ found that when increased quantities of gas were admitted, the pressure drop increased until the bed began to expand. When the gas velocity has increased to a point such that the pressure across the bed equals the weight of the bed per unit cross-sectional area, the bed expands. The bed then, to a certain extent, expands further with increasing gas velocities, with the pressure drop remaining constant^(22,52), as shown in Figure 2, page 29.

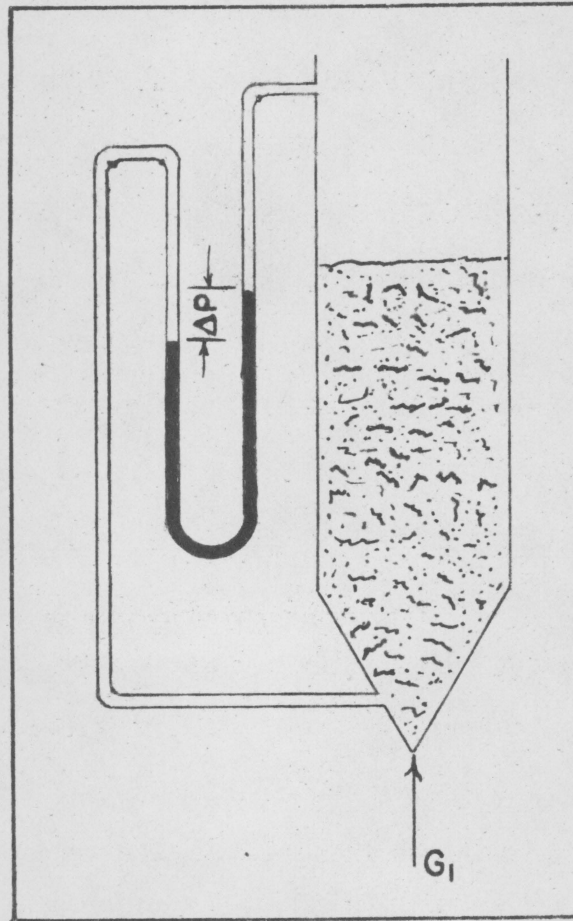


FIGURE 2. BED OF SOLID PARTICLES
PRIOR TO FLUIDIZATION.

LEVA, M. ET AL: INTRODUCTION TO FLUIDIZATION,
CHEM ENG PROG., 44, 511 (1948).

Patterson⁽⁹¹⁾ stated that free fall or classification of the particles may take place at less than fluidization velocities of the gas. Lewis⁽⁶⁰⁾ found that if in an expanded bed the velocity of the gas is decreased, the bed height decreases and a point is reached at which the bed appears to be quiescent. In this quiescent state the characteristics of the bed are very similar to those of quicksand, that is, each particle is separated from the other by a film of fluid; but the gas velocities are not sufficient to impart any significant turbulent motion to the particles.

Behavior of a Bed of Solid Particles at Minimum Fluidisation Velocity. Leva^(53,48) determined that before fluidization could take place a certain amount of bed expansion was required, and that this was entirely dependent upon the original static bed density. Leva⁽⁵³⁾, Matheson⁽⁶¹⁾, and Mørse⁽⁸²⁾ found that fluidization begins when the gas velocity is sufficient to expand the bed such that the solid particles become separated enough to permit individual or internal particle motion as shown in Figure 3, page 31. The velocity of the fluid required to separate the solid particles in a bed, such that internal particle motion exists, is known as minimum fluidization velocity.

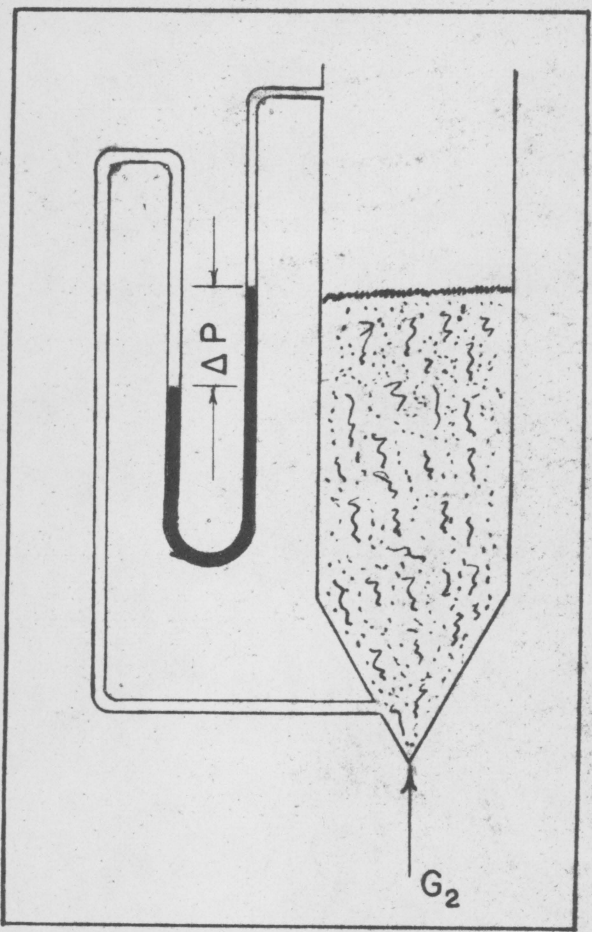


FIGURE 3. FLUIDIZED BED OF SOLID PARTICLES.

LEVA, M. ET AL: INTRODUCTION TO FLUIDIZATION
CHEM. ENG. PROG., 44, 511 (1948).

Behavior of a Bed of Solid Particles at Greater than Minimum Fluidization Velocity. Ergun and Orning^(21,22), and Matheson⁽⁶¹⁾ found that further increases in gas velocity above the minimum fluidization velocity caused a discontinuous flow of gas through the bed, approaching the point of a single dilute phase. Ergun and Orning determined that there was only a slight circulation of particles within the bed until bubbling flow was present. Leva⁽⁵³⁾ found that as gas velocities were increased above the minimum fluidization velocity, the bed expands further and that particle as well as bed motion is intensified, as shown in Figure 4, page 33. Leva noted that the bed motion resembled that of water being boiled. Matheson, Patterson⁽⁹²⁾, and Leva⁽⁵³⁾ noted fine particles being entrained above the fluidized bed.

The Types of Fluidized Beds. Lewis⁽⁵⁷⁾, for the purpose of experimental procedure, classified beds into batch and continuous fluidized operations. In batch operation no effort was made to return entrained particles to the bed; while in continuous operation, the solid particles moved continuously through the fluidization unit. In operation of continuous units, the Standard Oil Company of New Jersey and the Standard Oil Development Company⁽¹⁸⁾ classified fluidized beds according to their use and density. This classification of fluidized

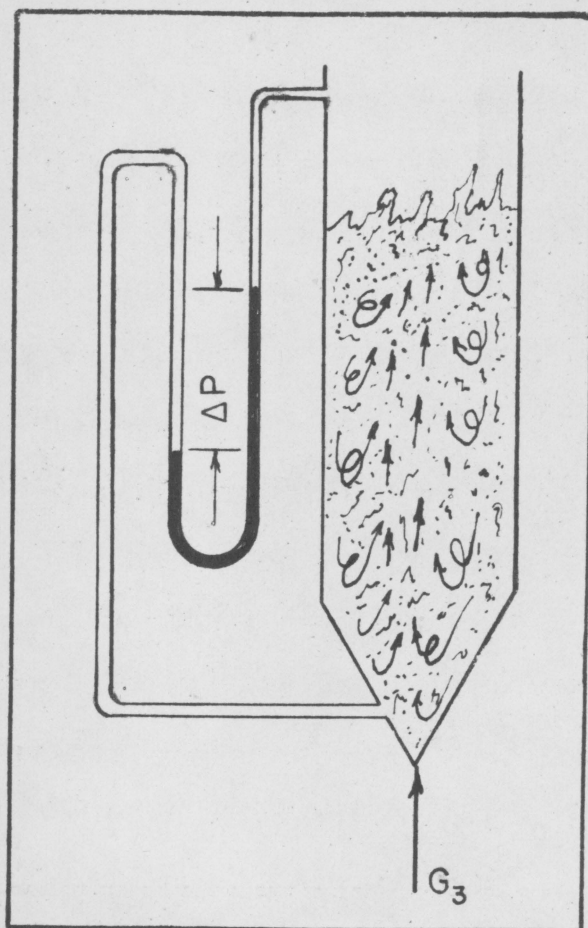


FIGURE 4. INTENSELY FLUIDIZED BED
OF SOLID PARTICLES.

LEVA, M. ET AL: INTRODUCTION TO FLUIDIZATION,
CHEM. ENG. PROG., 44, 511 (1948).

beds were, extremely dense (60 to 90 per cent of settled bulk), medium dense (5 to 60 per cent of settled bulk), and low density (about 5 per cent of settled bulk).

The Types of Dense Phase Flow. Matheson^(61,62) observed that three types of flow existed in the dense phase, (1) a cohesive type, (2) the aggregative type with good fluidisation, and (3) the slugging type. The flow characteristics of the dense phase were found to be influenced by the characteristics of the solid, the gas, and by the bed dimensions. Cohesive flow was primarily encountered with very small particles. Using synthetic cracking catalyst, 10 microns or smaller, Matheson found that cohesion was so great that severe balling of the solids resulted. With somewhat larger particles of the same material, in the range up to about 25 microns, cohesion still controls the bed appearance but balling is not in evidence. When the size of the catalyst was increased to about 40 microns, Matheson observed that the character of the flow becomes that of good fluidisation. If the velocity of the gas is increased, expansion of the bed takes place and a certain degree of freedom to move is imparted to each particle. The expansion of the bed continues in a uniform manner with increasing fluid velocity until apparently the rate of gas flow is greater than that which can percolate through the voids of the solid bed. Matheson and Leva⁽⁴⁹⁾ observed that at this stage bubbles form to

carry the excess gas from the bed, and as these bubbles grow in size they pass through the bed by coalescing with other bubbles. When the size of the bubbles attain the diameter of the vessel, "slug flow" is obtained. Matheson concludes that vessel diameter and bed depth have a direct relationship with respect to slugging. Morse⁽⁸²⁾ described "slug flow" as the entire bed of solid particles being lifted by the gas stream, like a piston, forming an arch from wall to wall of the vessel. In the majority of the cases the arch will break away, and the underside will fall in clumps or aggregates. Leva⁽⁵³⁾ also observed "slug flow", and stated that it was accompanied by large pressure fluctuations; while in aggregative fluidization very little pressure fluctuations were noted.

Characteristics of a Dense Normally Fluidized Bed. In the following discussion, the important characteristics of a dense normally fluidized bed such as quality of the bed, density of the bed, pressure drop across the system, the relation of pressure to the velocity of the fluid, viscosity of the solids, contact time in the vessel, mixing, particle entrainment, heat transfer in the fluidized bed, temperature control of the bed, and the ease of solids transfer are reviewed.

General Description of Dense, Normally Fluidized Bed. According to Matheson⁽⁶²⁾, a bed of normally fluidized particles consists of a gas phase rising, in

the form of bubbles, through a fluid consisting of solid particles by a small fraction of the total amount of the gas passing through the bed. Campbell⁽⁹⁾ described the system as a pseudoliquid phase in which the solid particles are completely air-borne, remaining relatively close to each other in a concentrated mixture, flowing in an eddying fashion. Ergun and Orning⁽²²⁾ and Matheson⁽⁶¹⁾ described the system as consisting of two phases, a liquid-like dense phase, and a relatively dilute suspension of solids entrained above the dense phase. Murphree⁽⁸⁵⁾, Matheson⁽⁶¹⁾, and Patterson⁽⁹¹⁾ further described the fluid bed, which was normally fluidized, as resembling a boiling liquid, because all of the particles present were in violent turbulent motion.

Quality of Fluidization. Morse⁽⁸¹⁾ has defined quality of fluidization to mean the uniformity of particle dispersion and the uniformity of gas velocity in the turbulent bed. The more uniform these factors, the higher the quality of fluidization. Water-fluidized beds of sand are considered to approach perfect quality, while beds in which slugging and channeling take place readily are said to be of the poorest quality. Morse further stated that the bed in which the gas pockets remain small and well distributed, with a steady, even,

overall boiling of the bed, is intermediate in uniformity, but it is perhaps optimum in the balance between uniformity and the requirement of mixing and turbulence in order to obtain temperature control.

Degree of Segregation of Particles in a Fluidized Bed. Morse^(81,82) pointed out that the basic difference between gas and liquid fluidized beds was the degree of segregation of the solid particles from the fluidizing medium. A highly segregated bed contains comparatively long channels and large clumps of only slightly separated particles, and the fluid rushing through the channels tears particles from the lower clumps and deposits the particles at subsequent low-velocity pockets or at the surface of the bed. The upper clumps descend as the lower ones are torn apart, thus completing the circulation path. Morse states that on the other hand, in a bed of slight segregation the channels are short, small, and well distributed throughout the bed, with the particles in the surrounding clumps separated from one another to such an extent that the path of flow through the clumps is not of much higher resistance than the path through the channels. Thus, the fluid flow is much more uniformly distributed, the fluid flow velocities smaller, and the particle circulation is primarily local.

Density of the Fluidized Bed. Matheson⁽⁶³⁾ states that bed density of fluid-solid systems is of interest

both from the fundamental standpoint and engineering considerations. The maximum bed density is obtained at the velocity at which the pressure drop over the measured portion of the bed is a maximum with the bed aerated and a few bubbles rising. The bed density represents the density of the continuous phase of the bed and is analogous to the density of a true liquid. Matheson states that a decrease in bed density with increasing gas velocity is caused by the presence of gas bubbles in the bed, while Lewis⁽⁶⁰⁾ observed that at constant gas velocities there were considerable variations in bed depth and therefore variations in the average bed density.

Pressure Drop through a Fluidized Bed. Morse⁽⁸³⁾ noted that the pressure drop through the fluidized bed is less than through a corresponding fixed bed at the same gas velocity, or that at equal pressure drops more gas would flow through a fluidized bed.

Relation of Pressure Drop Through a Fluidized Bed to Velocity of the Fluidizing Medium. Zenz^(104,105) determined and plotted a schematic summary of particulate gas flow characteristics. The pressure drop per unit-length of bed was plotted against the superficial fluid velocity on a log-log scale, as in Figure 5, page 40. The curve ϵ_s , represents the fraction of voids in a

densely settled fixed bed, and was included for comparison with the parallel curve ϵ of which represents the fraction of voids in the loosest possible fixed bed. Zenz noted that at velocities through ϵ of the bed becomes fluidized, as it is at this point that the pressure drop becomes equal to the weight of the material suspended per unit cross-sectional area. It was observed by Zenz as well as Matheson⁽⁶²⁾ and Ergun and Orning⁽²¹⁾ that on increasing the flow the bed expands and the fractional voidage in the bed increases with a corresponding reduction in pressure drop. The fluidisation curve, in Figure 5, intersects the empty pipe pressure drop line at u_t , or the terminal velocity of the particles. It can readily be seen from Figure 5 that a small change in gas velocity, at high voidages, will result in a large change in pressure.

Viscosity of Fluid-Solid Systems. Matheson^(65,66) made visual observations and comparisons of the behavior of aerated liquids and fluidized solids. He concluded that the gas passes through small size solids of good fluidity in a manner analogous to the upward flow of a gas through liquids of low viscosity, and that aerated liquids show the same tendency to slug as do solids. Matheson measured the viscosity of the fluidized solids bed by means of a modified Stormer viscosity meter. The measurement is not however, true viscosity because



FIGURE 5. RELATION OF PRESSURE DROP TO FLUID VELOCITY IN A FLUIDIZED BED.

ZENZ, F. A.: TWO-PHASE FLUID-SOLID FLOW, IND. ENG. CHEM.,
41, 2804 (1949).

accelerations of masses is involved in addition to shear properties. Matheson found that the Stormer viscosity of "narrow-cut" solids increases as the particle size and particle density is increased. He defined the viscosity of the fluidized bed as the Stormer net weight in grams, determined with a superficial air velocity through the bed slightly higher than that at which the Stormer torque becomes independent of the air velocity.

Mixing in Fluidized Solids Systems. Patterson⁽⁹¹⁾ found that because of the violent agitation of the solid particles, mixing within the bed may be considered instantaneous. In the analyses of solids drawn from various locations in the fluidized bed, very little variation was shown in the distribution of the particle sizes. Morse⁽⁸⁰⁾ noted that the advantage of this mixing action was the reduction of uneven temperature distribution.

Heat Transfer and Temperature Control in the Fluid-Solid Bed. Morse⁽⁸⁰⁾ and Mickley and Trilling⁽⁷⁸⁾ found that the fundamental advantage of the fluidized solids system is its ability to maintain a uniform temperature distribution throughout the bed. Along with Murphree⁽⁸⁶⁾ they concluded that the rapid circulation of solids within the vessel tends to give a uniform temperature throughout the mass. Patterson⁽⁹²⁾ stated that an

essentially instantaneous rate of heat transfer existed between the solids and the gas phase, because of the high surface to mass ratio. Campbell⁽¹²⁾ came to the same conclusions, stating that the entering air comes almost immediately to bed temperature. Murphree⁽⁸⁵⁾ and Patterson⁽⁹²⁾ stated that as a result of very uniform temperature distribution, temperature control within the bed is facilitated. Matheson⁽⁶³⁾, because of the extreme turbulence existing in the fluid bed, reasoned that heat transfer to and from the bed was excellent, as did Patterson⁽⁹²⁾ who found that heat transfer rates from the wall of the vessel to the bed of fluidized solids about the same as those for liquids in contact with the vessel walls. In studies of heat transfer in externally heated fluidized beds, Mickley and Trilling⁽⁷⁸⁾ obtained coefficients ranging from 3 to 70 times as great as those obtained with the same gas flow rates but in the absence of particles. Brecken⁽⁷⁾ obtained coefficients ranging from 1.5 to 45 times as great as those obtained with the same gas flow rates in the absence of particles. For very low gas velocities, Parent⁽⁹⁰⁾ obtained coefficients of heat transfer three per cent lower for fluidized beds than were obtained for empty vessels, however when the gas velocities were increased he obtained heat transfer coefficients about

five per cent higher for the fluidized beds than for the empty reactors. Campbell⁽¹¹⁾ and Murphree⁽⁸⁶⁾ state that the solid within the reactor impart heat capacity to the system, and Mickley and Trilling⁽⁷⁸⁾ noted that this heat capacity is important in close temperature control of reactor vessels.

Mickley and Trilling⁽⁷⁸⁾ have summarized the following methods for temperature control in a fluidized reactor: (1) control of temperature and flow rate of the entering fluid, (2) control of the temperature and cycling rate of solid particles entering the system, (3) transfer of heat to heat exchanger surfaces within the retort, (4) transfer of heat to the walls of the container.

Ease of Solids Transfer by Fluidized Solids Technique. According to Hariu and Molsted⁽⁴²⁾, Leva⁽⁵²⁾, and Matheson⁽⁶¹⁾ the ease with which solids can be transferred from vessel to vessel, is an advantage of the system conducive to continuous process application. Matheson also reasoned that such a system permits the most efficient utilization of heat.

Variables Affecting the State of Fluidization. In order that correlations can be made between the yield of oil from retorting oil shale in a fluidized bed, and variables affecting fluidization, a knowledge of the properties of the materials

making up the fluidized system, and the effect of bed dimensions on the state of fluidization, is important.

Effect of Variables of the Fluidization Medium.

The effects of velocity, density, and viscosity of the fluidization medium on fluidization, are contained in the following paragraphs.

Velocity. Patterson⁽⁹²⁾ and Matheson⁽⁶³⁾ found that the bed density decreased with an increase in gas flow, and according to Patterson the maximum bed density is obtained at the minimum fluidization velocity. Matheson went further to describe the effect of varying velocities on the solids in the vessel. At first the gas percolates through the bed without agitating the particles, and as the velocity continued to increase, the bed expands until a certain amount of movement is noted among the particles. A single liquid-like phase exists at this point, but as the velocity is increased further, the particles of solid begin to entrain overhead, and a two-phase system is formed. As the velocity continues to increase the two phases become more alike until a velocity is reached at which total entrainment is encountered with a single dilute suspension existing. Leva⁽⁵³⁾ observed that channeling was more pronounced at low flow rates because violent agitation of the bed at high gas velocities destroyed the channels.

Patterson⁽⁹⁴⁾ found that the pressure drop through the bed was nearly independent of the gas velocity.

Density of the Fluidizing Medium. Patterson⁽⁹³⁾ states that the effect of the gas density within the ranges encountered in fluidization is a relatively minor factor. This is explained by the fact that the driving force required to maintain a fluidized bed is a function of the difference between the density of the solid and the density of the gas, and the gas density is generally negligible. Matheson⁽⁶⁴⁾ found that the effect of the gas density on the bed density was negligible and Lewis⁽⁵⁹⁾ found it to have no effect on the gas velocity.

Viscosity of the Fluidizing Medium. Matheson⁽⁶⁵⁾ found that the bed of maximum density increased in density with a decrease in gas viscosity, and Lewis⁽⁵⁹⁾ found that the velocity of the gas to be inversely proportional to the viscosity.

Effect of Solids Characteristics on Fluidization. The characteristics of solids which have been found to affect fluidization are discussed in the following paragraphs. Some of the more important characteristics which affect fluidization are diameter, density, shape, surface tension, and electrostatic charge.

Diameter of Particles. Leva⁽⁴⁹⁾ and Matheson⁽⁶³⁾ determined that the minimum fluid voidage, ϵ_{mf} ,

increased for a decrease in particle diameter. Ergun and Orning⁽²²⁾ found that the diameter of the particles, when increased, increase the channeling in the fluidized bed. As shown by Patterson⁽⁹³⁾ as the particle diameter is increased the gas flow rate must also be increased to maintain a given fluidized bed density. Leva⁽⁵⁴⁾ found that fine materials have the greatest tendency to channel, but Morse⁽⁸⁴⁾ found that an increase in particle size increased segregation. Matheson^(62,65) observed that the rate of bubble growth was directly dependent on the particle diameter, increasing particle diameter increased bubble growth. He also found that an increase in particle size increased the Storer viscosity of the fluidized bed.

Density of Particles. Patterson⁽⁹³⁾ found that for a given flowrate and particle diameter, the bed densities remain essentially constant, and varies very little with particle density, although Matheson⁽⁶³⁾ observed an increase in maximum bed density with particle density. Matheson also found that the rate of bubble growth increased with increased particle density. Morse⁽⁸⁴⁾ observed an increase of particle segregation with a corresponding increase in particle density. Lewis⁽⁵⁹⁾ found that the velocity of a gas necessary for fluidization was proportional to the density of the solid.

Shape of Particles. According to Patterson⁽⁹³⁾, very little is known about the effect of particle shape on fluidization. He reasoned from aerodynamic considerations, that smooth, rounded solid particles would be expected to require a somewhat higher gas velocity for fluidization than rough, irregular particles of the same size distribution and density. Leva⁽⁵⁵⁾ found that a decrease in fractional voids from 0.435 to 0.367, increased the pressure drop about 100 per cent. Leva⁽⁴⁸⁾ noted that any bed composed of minimum fluid voidage will begin to fluidize under the influence of very small fluid quantities, while beds with high fluid voidages large amounts of fluid are required before fluidization will take place. Seidel⁽⁹⁵⁾ studying particles of dissimilar shape, but with like bulk densities, found that there was no definite relationship between particle shape and pressure drop. Matheson⁽⁶⁵⁾ reported that the Störmer viscosity is much larger for spherical particles than for particles of irregular shape.

Surface Tension of Bed in Fluidized State.

Patterson⁽⁹³⁾ reported that surface tension is usually a minor factor in fluidization, except where very fine particles are involved. He further states that surface tension tends to aid fluidization, and that low surface tension on the particles tends to sometimes form

agglomerates of the solids in the fluidized bed, resulting in channeling of the fluidizing medium.

Electrostatic Charge on Particles. Patterson⁽⁹³⁾ found that electrostatic charge on the solid particles had very little effect on fluidization, but that a high electrostatic charge tends to facilitate fluidization. Particles which acquire a high electrostatic charge repel each other and thus require lower gas rates for fluidization.

Effect of Vessel Characteristics on Fluidization. The effect of vessel diameter and length on fluidization are not well known. Matheson⁽⁶²⁾ found that the larger the vessel diameter the less tendency there is for "slugging flow". Leva⁽⁵³⁾ observed that channeling took place in small diameter vessels more readily than in vessels of large diameter. Matheson⁽⁶³⁾ has shown that slugging can be reduced by decreasing the depth of the solids bed. Leva⁽⁴⁹⁾ found that the height of bed had no effect on maximum or minimum fluid voidage.

Mathematical Relationships for Fluidization. Some of the methods of calculating gas velocity for fluidization, pressure drop across a fluidized bed, and bed densities are presented in the following paragraphs, but as explained by Campbell⁽¹⁶⁾ many of the relationships that have been developed are reasonably satisfactory for some commercial uses, but are unsafe in universal application.

Gas Velocity Required for Fluidization.

Leva(56) derived the following equation for the mass flow of gas required for fluidization:

$$G = \frac{0.005 D_p^2 \epsilon^3 (\rho_s - \rho_f) \rho_f g}{\lambda^2 (1 - \epsilon) \mu} \quad (1)$$

where

G = mass superficial gas velocity, lb/hr-sq ft

D_p = average effective particle diameter, ft

ε = fraction voids in dumped bed, no dimensions

ρ_s = density of solid particles, lb/cu ft

ρ_f = density of fluid, lb/cu ft

g = acceleration due to gravity, 4.18 x 10⁸
ft/hr²

λ = particle shape factor, no dimensions

μ = viscosity of fluid, lb/hr-ft.

Leva(47) stated that the mass velocity required for the beginning of fluidization may be predicted by substituting the fraction voidage of the bed at minimum fluidization, ε_{mf}, for fraction voidage in dumped bed, ε, in Equation 1 above.

The general equation for gas flow rate at the beginning of fluidisation as given by Leva⁽⁴⁷⁾ is:

$$G_{mf}^2 = \frac{D_p g \rho_F (\rho_s - \rho_F) \epsilon_{mf}^3}{2 f \lambda^{3-n} (1 - \epsilon_{mf})^{2-n}} \quad (2)$$

where

G_{mf} = minimum mass superficial fluid velocity required for fluidisation, lb/hr-sq ft

D_p = average effective particle diameter, ft

g = acceleration due to gravity, 4.18×10^8 ft/hr²

ρ_F = density of fluid, lb/cu ft

ρ_s = density of particles, lb/cu ft

ϵ_{mf} = fraction voidage of bed at minimum fluidisation, no dimensions

f = modified friction factor, no dimensions

λ = particle shape factor, no dimensions

n = state-of-flow factor, no dimensions.

Bed Density. Matheson⁽⁶³⁾ found that for irregular particles, ranging in size from 28 to 456 microns in diameter and with bulk densities ranging from 27.5 to 134.0 pounds per cubic foot, the maximum bed density, ρ_{MB} , may be represented by:

$$\log D_p = 2.81 \frac{\rho_{MB}}{\rho_s} - 1 \quad (3)$$

where

D_p = average effective particle diameter, ft

ρ_{MB} = maximum bed density, lb/cu ft

ρ_s = density of particles, lb/cu ft.

Leva observed that smaller microspheres have maximum bed densities considerably greater than those observed for irregularly shaped particles in Equation 3.

Pressure Drop in Fluidised-Solids Systems.

Patterson^(92,94) found that within practical limits, the pressure drop through a fluidised bed is approximately equal to the static head of the bed in the following manner:

$$\Delta P = \frac{4W}{\pi D^2} = \rho_B L \quad (4)$$

where

P = pressure drop through the bed,
lb/sq ft

W = weight of solids in bed, lb

D = diameter of bed, ft

ρ_B = density of bed, lb/cu ft

L = depth of bed, ft.

Lewis⁽⁵⁸⁾ also concluded that the pressure drop through a fluidized bed was essentially equal to the weight of the solid particles and confined Equation 4 to solids of small diameters, and only beds with small ratios of length to diameter. Lewis found that Equation 4 did not hold for particles of diameters larger than 0.01-inch.

Hariu and Molstad⁽⁴³⁾ considered the total pressure drop as the sum of the pressure drop resulting from gas flowing through the empty vessel, and the solids pressure drop consisting of the static head and a friction loss factor resulting from particle-to-particle contact.

Wilhelm and Kwauk⁽¹⁰¹⁾ derived the following equation, based on the concept that fluidization takes place when the weight gradient of the solid bed is equal to the weight gradient through the bed:

$$\frac{\Delta P}{L} = (1 - \epsilon) (\rho_s - \rho_f) \quad (5)$$

where

$\frac{\Delta P}{L}$ = pressure gradient, lb/sq ft-ft

$1 - \epsilon$ = fraction of solid at height L

ρ_s = density of particles, lb/cu ft

ρ_f = density of fluid, lb/cu ft.

Leva (44,46,48,50) developed a formula similar to Equation 5, employing area of the bed and not height:

$$P = \frac{V_T}{A_T} (1 - \epsilon) (\rho_s - \rho_F) \quad (6)$$

where

P = pressure drop through bed, lb/sq ft

V_T = volume of dumped bed, cu ft

A_T = cross-sectional area of bed, sq ft

ϵ = fractional voidage in dumped bed

ρ_s = density of particles, lb/cu ft

ρ_F = density of fluid, lb/cu ft.

Application of Fluidisation

In the following paragraphs a review of the history, present commercial applications, possible future applications, limitations and advantages of the fluidized bed are given. Leva (52) and Patterson (91) regarded fluidization technique as a separate unit operations. Murphree (89) believed that the fluidized bed has wide application in both catalytic and non-catalytic industrial operations, and Tarber (25) states that the use of the fluidized bed in industrial application of Fischer-Tropsch reactions, which were previously too complex and economically unfeasible for commercial applications, a very attractive investment.

History of Applications of Fluidization

According to Murphree⁽⁸⁹⁾, the basic principles of the fluid solids technique were developed just prior to World War II with the catalytic cracking of petroleum as the immediate objective, but with the war providing the necessary impetus for this application but hindering developments in other fields in which the technique is proving highly useful. Matheson⁽⁶¹⁾ and Campbell⁽⁸⁾ also noted that the first successful commercial application of fluidisation was a unit for the catalytic cracking of petroleum early in 1942. Patterson⁽⁹¹⁾ stated that the development of fluidisation technique for commercial use has been primarily in the field of petroleum. Matheson⁽⁶¹⁾ gave production figures indicating an increase of fluid cracking capacity of 960,000 barrels per day over the 40,000 barrels per day in 1942. Gisher⁽³³⁾ applied the fluidized solids technique to the recovery of oil from tar sands in 1946, while Blanding and Roetheli⁽⁴⁾ applied the technique to the recovery of oil from shale in 1943.

Commercial Application of Fluidization. Campbell⁽⁸⁾ and Murphree⁽⁸⁸⁾ stated that the largest single application of fluidization on the commercial scale was that of the catalytic cracking of crude petroleum for the production of gasoline. Campbell⁽⁶⁾ lists the products of crude petroleum as high octane gasoline, butenes and isobutenes

for the production of high octane gasoline by alkylation, and isobutene and butadiene for the production of synthetic rubber.

Future Applications of Fluidization. Patterson⁽⁹¹⁾

gave the following as the operations to which the fluidized solids technique may be applied: (1) no chemical change in either the solid or gas, such as in heat transfer or the drying of the solids or gases; (2) no chemical change in the solids, such as gas reacting in contact catalysis, cracking, and oxidation processes; (3) no chemical change in gas, as in solids reacting for calcination; and (4) both solid and gas reacting, as in ore roasting and halogenation. Campbell^(8,10) listed other possible uses of the fluidized solids technique in future commercial applications as:

(1) devolatilization of wood; (2) calcination; (3) roasting; (4) mixing of solids; (5) drying of solids; (6) separation and purification of gases by solid absorbents and/or reactants; (7) recovery of vapors from gases; and (8) catalytic processes such as alkylation. Campbell⁽⁹⁾, Leva⁽⁴⁴⁾, Lewis⁽⁵⁷⁾, and Patterson⁽⁹¹⁾ gave several specific possible industrial applications which were under consideration, some of which are: (1) synthesis of liquid hydrocarbons from natural gas; (2) gasification of coal for the recovery of hydrocarbons; (3) production of phthalic anhydride by the oxidation of naphthalene; and (4) recovery of oil from oil shale.

Advantages of the Fluidized Bed Over Fixed Bed Reactors.

Leva⁽⁵³⁾ listed the following advantages of the fluidized bed over the fixed bed operation: (1) better temperature equalization; (2) better heat transfer within the bed; (3) lower mechanical energy requirements; and (4) simple equipment construction.

Limitations of the Fluidized Solids Technique.

Patterson⁽⁹¹⁾ gave the limitations of the fluidized solids technique as: (1) short gas to solid contact time; (2) lack of control over the average retention time of solids in the bed; and (3) limited flexibility of the operation because of necessity of keeping the gas flow within narrow limits.

III. EXPERIMENTAL

The experimental section on the retorting of oil shale by the use of the fluidized-solids technique includes the purpose of the investigation, the plan of experimentation, the materials and apparatus used, the procedure of operation, the data and results obtained, and the sample calculations.

Purpose of Investigation

It was the purpose of this investigation to evaluate the use of the fluidized-solids technique for the retorting of oil shale, and to determine the effect of oil shale particle size on the characterization factor and yield of shale oil.

Plan of Investigation

The plan of the investigation consisted of a survey of the literature, the design and construction of a fluidization unit, the experimental procedure, and the evaluation of the results.

Literature Survey. A literature search was made to obtain a general introduction to the field of oil shale applicable to the study of oil shale fluidization, to become familiar with the structure and properties of available oil

shale, to review the fundamental principles of fluidization, and to summarize the applications of the fluidized-solids technique.

Design and Construction of Fluidization Unit. The fluidization unit, designed and constructed for this investigation, consisted of a fluidization column, disengaging section, cyclone separator or knock-out tank, gas-heating section, gas circulating pump, two gas-cooled condensers, a water-cooled condenser, a brine-cooled condenser, a surge tank, gas-flow measuring and pressure recording equipment, a brine circulation system, and temperature control and measuring equipment.

Fluidization Column. The fluidization column assembly consisted of an insulated vertical fluidization column with thermocouple installations for measuring the bed temperatures, and manometer taps for obtaining the pressure differential over various sections of the fluidized bed. A distribution plate assembly was attached to the lower section of this column.

Gas-heating Section. The fluidization gas was heated electrically in a packed gas-heating section. The section had electrical heaters inserted through the wall with the electrical leads passing through the wall of the section. The heat to the section was controlled by means of a capacitrol operating

from a thermocouple placed immediately above the distribution plate in the fluidized bed. Supplementary heaters were attached directly to the gas transfer line from the gas-heating section to the fluidization column.

Disengaging Section. The disengaging section was attached directly to the top of the fluidization unit and designed to separate the major portion of entrained particles from the fluidizing gas. The cross-sectional area of the disengaging section was made four times that of the fluidization column and connected to the fluidization column by means of flanged reducers. The disengaging section was heated to prevent condensation of the shale-oil vapors on the wall of the section.

Cyclone Separator. The cyclone separator was attached to the disengaging section, and was designed to remove particles of solids which were not removed in the disengaging section. External heaters were attached to the cyclone to prevent the condensation of the shale-oil vapors on the wall of the cyclone separator.

Condensers. In order to condense the shale-oil vapors without plugging the condenser tubes by condensation of the asphalatic compounds, the shale oil was condensed in fractions. The first and second

condensers were gas-cooled. The first condenser was designed to operate at a minimum temperature of 250 °F, and the second condenser was designed to operate at a minimum temperature of 140 °F. The third, a water-cooled condenser, was designed to operate at a minimum temperature of 60 °F, and the fourth, a brine-cooled condenser, was designed to operate at 30 °F. The condensers were connected in series and the gases from the brine-cooled condenser were used as the cooling medium in the first and second condensers. The condensers were simple double-pipe type exchangers with the cooling media passing through the annular space.

Surge Tank. The surge tank was designed to give the circulation pump a reserve for gas circulation. Return gas from condensers was brought into the tank at the top and the gas to the pump taken out immediately below the gas inlet. An atmospheric blow-off line was placed in the top of the tank for venting gases to the atmosphere. A blow-down line was placed in the center of the bottom of the tank so that any condensed material could be removed from the tank.

Gas-flow Measuring and Pressure Recording Equipment. Gas flow was measured by means of a calibrated orifice with manometer. The pressure of the system was recorded on a round chart recorder. The pressure drop across the

fluidized bed was measured by means of manometers, the leads of which were attached at intervals along the height of the bed.

Brine Circulation System. A brine-cooled condenser was employed to condense some of the gases leaving the water-cooled condenser. The brine was circulated through the condenser from an ice-salt mixture maintained in a storage tank.

Temperature Control and Measuring Equipment.

The temperature of the fluidizing gas was measured and controlled by a mechanical potentiometer and stepwise controller. Temperatures over the entire system were measured by means of iron-constantan thermocouples in conjunction with an interlocked-pushbutton potentiometer. Temperatures in the cyclone was controlled by adjustable rheostats and an on-off controller operating from a thermocouple in this unit. The temperature of the disengaging section was controlled by adjusting rheostats when the desired temperature was indicated by the thermocouple measuring the temperature. All thermocouples were calibrated in position.

Experimental Procedure. The fluidized bed consisted of oil shale as the solid and carbon dioxide as the fluidizing medium. Tests were made on solids of four sizes, using gas

flow rates as required for fluidisation. The retort of the fluidizing unit was charged with oil shale of the desired size and the system purged with carbon dioxide. The system was then pressurized. The gas circulation was then begun and all heaters and auxiliary equipment were put into operation. A test was complete after retorting for one hour at 715 °F.

The volume of gases generated by retorting of oil shale was taken as the difference between the volume of gas added and the volume of gas in the retorting unit after retorting. The shale oil was collected and its characterization factor determined. The composition of retort gases were determined. The retorted shale was collected and its shale oil content determined.

Evaluation of Results. The results of the retorting test were evaluated by comparing the characterization factor, the volume of gases generated, and the yield of shale oil as functions of the particle size of the shale retorted.

Materials

The following materials were used in this investigation:

Acid, Hydrochloric. Analytical reagent, C. P., catalog number A-114. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used as an analytical reagent in determining the carbon monoxide in the gases of the system.

Acid, Pyrogallic. Crystal, reagent grade, lot No C113j, code No 1186. Manufactured by General Chemical Division, Allied Chemical and Dye Corp., New York, N. Y. Used in a solution with potassium hydroxide for the determination of the oxygen in the fluidization gas.

Benzene. Technical grade, 99 to 100 per cent pure, lot number 8240, code No 1444. Manufactured by General Chemical Division, Allied Chemical and Dye Corp., New York, N. Y. Used as a solvent for cleaning the equipment before tests.

Calcium Chloride. Flake, 77-80 per cent purity, lot number E308j, code No 1518. Manufactured by General Chemical Division, Allied Chemical and Dye Corp., New York, N. Y. Used as a drying agent in analysis of retort gases.

Carbon Dioxide. Commercial grade, 99.0% purity, 50-pound cylinder. Obtained from Southern Oxygen Company, Roanoke, Virginia. Used as the fluidizing medium in fluidisation system.

Cuprous Chloride. Powder, reagent grade, lot No ED18, code 1656. Manufactured by General Chemical Division, Allied

Chemical and Dye Corp., New York, N. Y. Used in conjunction with hydrochloric acid in orsat gas analysis apparatus for absorbing carbon monoxide.

Cupric Sulphate. Crystal, reagent grade, lot No 45, code 1649. Manufactured by General Chemical Division, Allied Chemical and Dye Corp., New York, N. Y. Used to determine the hydrogen sulphide in the retort gases.

Dibutyl Phthalate. Normal purified. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used as manometer fluid.

Grease, Stopcock. Silicone, Dow-Corning. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to grease stopcocks on Orsat apparatus.

Grease, Waterproof. Essoleum. Obtained from Esso Standard Oil Company, Radford, Virginia. Used as shaft lubrication for gas pump.

Mercury. Purified. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used as manometer fluid.

Oil. No 30 SAE. Obtained from Esso Standard Oil Company, Radford, Virginia. Used as lubricant in gas pump.

Oil Shale. Containing approximately 20 per cent distillable organic matter. Obtained from the United States Bureau of Mines, Rifle, Colorado. Taken from the Mahogany Ledge. Used as the test material in the fluidized bed.

Potassium Hydroxide. Pellet, Lot No 0283j, code 2256. Manufactured by General Chemical Division, Allied Chemical and Dye Corp., New York, N. Y. Used as an absorbent for carbon dioxide in orsat apparatus.

Apparatus

The following apparatus was used in this investigation:

Balance, Analytical. Chain-o-matic, capacity 100 grams, sensitivity \pm 0.0002 gram, model No 220-D, serial M-1-13860. Manufactured by Volland and Sons, Inc., New Rochelle, N. Y. Used in the analysis of shale oil.

Balance, Triple-Beam. Laboratory type, capacity 0 to 21 kilograms, graduated in grams and tenths of grams. Manufactured by Ohaus Company, Newark, N. J. Used to weigh oil shale for the fluidized bed.

Beakers. Pyrex, Griffin, with spout, miscellaneous sizes. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in handling shale, manometer fluids, and shale oil.

Bottles, Gas Sample. Pyrex, 250-milliliter capacity, with stopcocks. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used for sampling gases of fluidization system.

Bottles, Sample. Pyrex, water sample, with wide mouth and flat headed ground stoppers, capacity 125 milliliters.

Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to store shale oil samples.

Bottle, Specific Gravity. Gay-Lussac, pyrex, ten milliliters with perforated glass stoppers, ground flat on two sides. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to determine the specific gravity of shale oil.

Combs Gyrotory Riddle and Motor. Made by the Great Western Manufacturing Company, Leavenworth, Kansas. Used for shaking screens, in preparing oil shale for retorting.

Crucibles. Coors, porcelain, high form, with covers. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in the determination of oil shale moisture.

Desiccator. Type F and S, diameter inside 250 milliliters. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to store oil shale samples before weighing.

Distillation Apparatus. ASTM distillation apparatus, according to ASTM D86. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to determine the 50 per cent point of shale oil.

Extraction Apparatus. Soxhlet, pyrex with 24/40 T ground joints, with side arm and condenser. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to remove the solids from the shale oil and to recover the solvent.

Flask, Volumetric. Glass stoppered, blue line exax, with stoppers. Capacity 250, 500, and 1000 milliliters. Used for making analysis solutions.

Fluidization Unit and Accessory Equipment. All materials required for the construction and operation of the fluidization unit and accessory equipment are included in Table V of the Appendix.

Funnel, Filtering. Pyrex glass, 58° angle, long stem. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in the analysis of the shale oil.

Gas Meter. Precision wet test meter, graduated in 1/10th cubic feet. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to determine the volume of fluidization system.

Graduated Cylinder. Blue line exax. Capacity 25, 50, 100, and 500 milliliters. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used for measuring shale oil samples and for holding liquids for use in gas sample bottles.

Jaw Crusher and Motor. Serial No 101. Obtained from Universal Crusher Company, Grand Rapids, Michigan. Used to crush oil shale.

Kinematic Viscosity Apparatus. The apparatus for measuring the kinematic viscosity of the shale oil of a constant temperature bath, and an ostwald viscosity pipet,

Fenske modification. Numbers 200, 300, and 400 exax. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used for determining the viscosity of shale oil.

Miscellaneous Equipment. Drill press, drills, welding equipment, pipe threaders, test meters, welding rod, silver solder, flux, cutting oil, hack saws, wrenches, and other equipment and necessary tools required for the construction of the equipment. Obtained from Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia, and privately owned tools.

Orsat Gas Analysis. Fisher unitized, technical universal model. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to analyze retorting gases.

Rod Mill. Patterson ball mill. Installed in Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used with 10 pounds of rods, to reduce the size of oil shale.

Sample Splitter. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in quartering oil shale samples and obtaining representative oil shale sample for shale oil analysis.

Sieves. Standard Tyler. Mesh sizes 4, 6, 8, 14, 16, 20, 28, 40, 48, 100, 150, 200, 250, and 325, pan and screen top included. Manufactured by W. S. Tyler Company, Cleveland,

Ohio. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in sizing of oil shale for fluidisation and solids remaining in retort after retorting.

Timer. Electric, 115 volts, 60 cycle, 5 watts, graduated in seconds, serial No 19605, catalog No 14-653. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used for timing experimental tests.

Method of Procedure

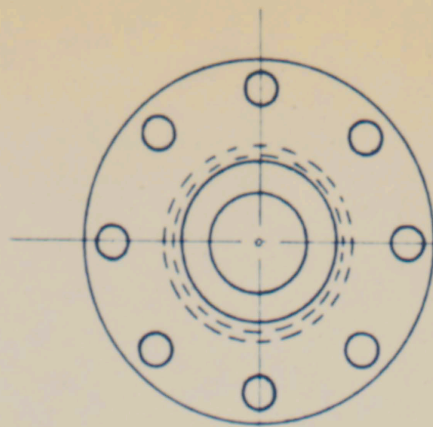
The procedure followed in carrying out this investigation included the design and construction of the fluidization unit, calibration of orifice and thermocouples, observation of fluidization characteristics of oil shale, operation of fluidization unit, and analysis of oil shale, shale oil, residue of retort, and retort gases.

Design and Construction of Fluidization Unit. The first consideration in the design of the retorting unit was the length and diameter of the fluidization chamber, and the design of the gas-heating section. The following paragraphs are detailed descriptions of the design and construction of the fluidization chamber, gas-heating section, cyclone separator, condensers, surge tank, gas-pumping section, gas-measuring section, and control units.

Design and Construction of Fluidization Chamber.

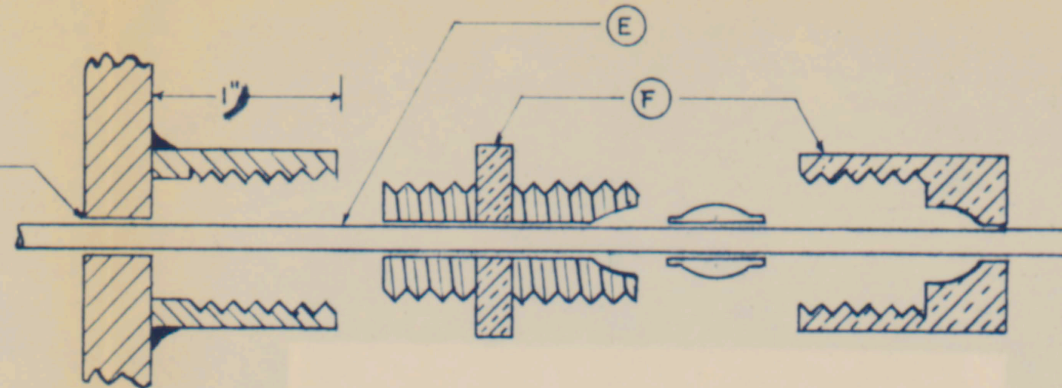
The fluidization section, as shown in Figure 6, page 71, was constructed from a 36-inch section of 4-inch nominal diameter black iron pipe. The inner wall surface was swabbed clean with benzene and rinsed. In order to determine the pressure drop over the fluidized bed, manometer taps were made 3 inches above the distribution plate, 21 inches above the distribution plate, and 3 inches below the disengaging section. The manometer taps were made by welding

Figure 6
Heating Section

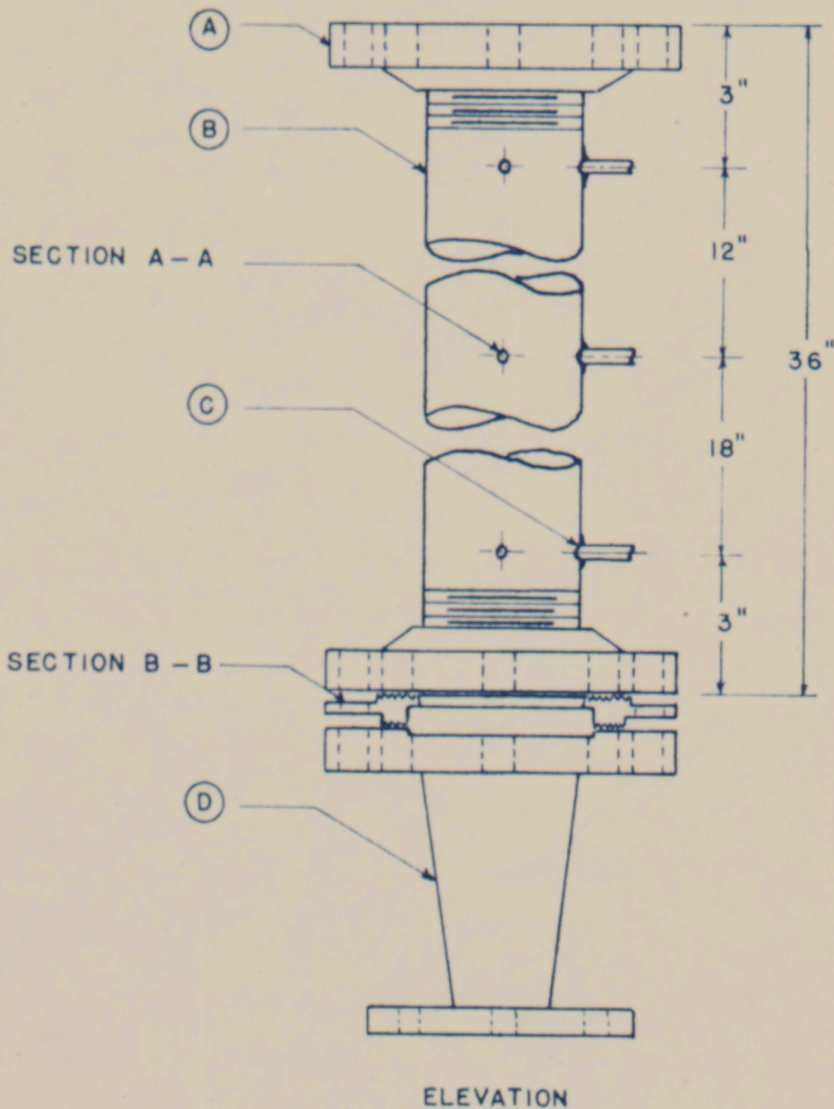


PLAN

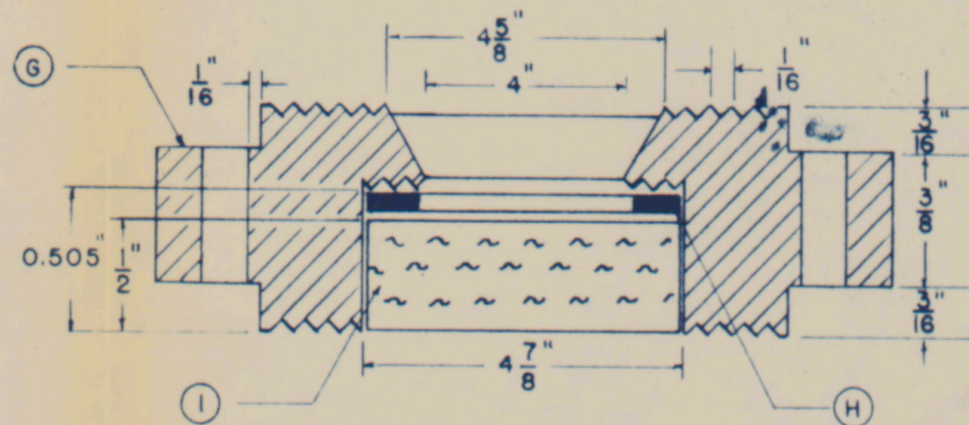
$\frac{3}{16}$ " DRILL



SECTION A - A



ELEVATION



CROSS SECTION B - B

LEGEND

- (A) = STANDARD 4-IN. CAST IRON FLANGE
- (B) = 4-IN. STEEL PIPE
- (C) = $\frac{1}{8}$ -IN. WELDED MANOMETER TAPS, BLACK IRON COUPLINGS USED
- (D) = STANDARD 4 X 2 $\frac{1}{2}$ -IN. STEEL REDUCER
- (E) = $\frac{1}{8}$ -IN. DIAM. IRON-CONSTANTAN THERMOCOUPLE
- (F) = FERRULE FITTING $\frac{1}{8}$ -IN. PIPE TO $\frac{1}{8}$ -IN. COPPER TUBING ADAPTER
- (G) = STANDARD 4-IN. DEAD FLANGE, MACHINED TO SPECIFICATIONS OF SECTION B-B, WITH CONCENTRIC RINGS $\frac{1}{8}$ -IN. DEEP
- (H) = STANDARD 4-IN. ASBESTOS GASKET
- (I) = FUSED SILICA, FILTROS PLATE GRADE "A"

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RETORT AND
DISTRIBUTION PLATE

SCALE: NONE DATE 2/3/55 CASE NO:
DRAWN BY: T. G. WIGANO FILE NO:
CHECKED BY: Jesse M. Carr, Jr. FIGURE NO: 6
APPROVED BY: 408 4/12/55 SHEET NO:

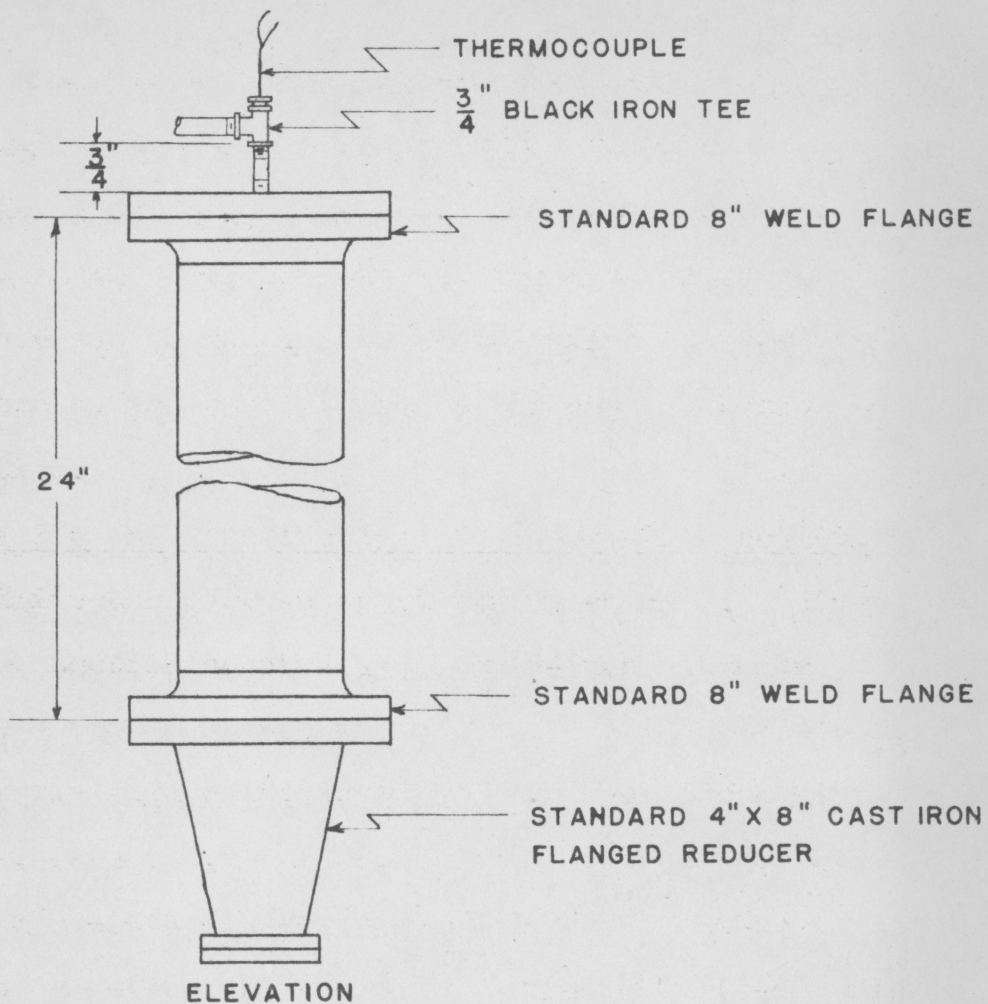
1/8-inch black iron high pressure couples to the fluidization chamber wall, and then drilling five 1/32-inch holes through the pipe wall inside the couple. This afforded a transfer of pressure but prevented solids of the fluidized bed from clogging the manometer lines. Thermocouples were placed on the same levels as the manometer taps by means of ferrule fittings in 1/8-inch black iron high pressure couples. The thermocouple lengths were adjusted so that the thermocouples immediately below the disengaging section and above the distribution plate were in the center of the fluidized bed. The thermocouple at the 21-inch bed level was placed at the bed wall.

The distribution plate at the bottom of the fluidization column consisted of a 4-inch cast iron flange lathed to the dimensions as shown in Figure 6, page 71, cross section B-B. A "Filtros" plate 0.50 inch thick was used as the main fluidizing plate, with a quarter inch steel plate with holes drilled 1/8-inch diameter on 3/32-inch centers used as a supporting plate. The supporting plate was counter-sunk into the face of the 4 by 2-1/2-inch flanged reducer. The "Filtros" plate was gasketed between the lathed flange to prevent the passage of gas around the edges of the plate. The

distribution plate, the 2-1/2-inch by 4-inch flanged reducer, and the 4-inch retort was assembled into one unit as shown in Figure 6. The unit was loaded and unloaded by removing the entire assembly from the fluidization unit. The fluidization chamber was insulated by use of standard 85 per cent magnesia insulation.

Design and Construction of Disengaging Section.

To minimize the carry-over of fine particles, a disengaging section constructed from 8-inch diameter pipe, was placed at the top of the fluidizing chamber, as shown in Figure 14, in appendix. The disengaging section, Figure 7, page 74, was constructed from a 4-inch by 8-inch flanged reducer joined to a two foot section of 8-inch pipe by means of 8-inch slip-on weld flanges. The disengaging section was terminated by means of bolting an 8-inch diameter cast iron blind flange to the top welded flange of the disengaging section. The cast iron blind flange was tapped 3/4-inch pipe size in the center to provide an outlet for the fluidizing gas and oil vapors. In order to prevent condensation of the oil vapors on the inside surface of the disengaging section, surface heaters of 1000 watts capacity in ceramic plates were attached to the outside wall by means of studs welded to the pipe wall.



NOTES

- ① DISENGAGING SECTION ATTACHED TO THE TOP OF FLUIDIZED RETORT
- ② INSULATION AND HEATERS NOT SHOWN

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DISENGAGING SECTION

SCALE: NONE DATE 4/10/55 CASE NO:
DRAWN BY: T. G. Vignani FILE NO:
CHECKED BY: J. M. C. 4/12/55 FIGURE NO:
APPROVED BY: JWB 4/12/55 SHEET NO:

Three heaters were placed in a circle, six, ten, and fourteen inches below the top of the disengaging section. The disengaging section was gasketed with corrugated asbestos-steel gaskets, and was insulated with standard 8-inch, 85 per cent magnesia insulation.

Gas-heating Section. In order that coking would not occur on the retort wall, and to give even heat transfer through the fluidized bed, the fluidizing gas was heated to the desired retorting temperature before passing the gas into the fluidized bed. The heating of the fluidizing gas was accomplished by passing the gas through a gas-heating section constructed from a five-foot section of eight-inch diameter black iron pipe with weld-neck flanges attached to each end. As shown in Figure 8, page 76, the heating elements were placed inside the eight-inch pipe, and the wiring leads were inserted through the pipe wall inside 1/2-inch high pressure couples which were welded into the pipe wall. The 1/2-inch couples were placed six inches apart, alternately on opposite sides of the pipe, beginning 12 inches above the face of the bottom flange. Before welding into the heating section, the 1/2-inch couples had 1/2-inch nipples screwed into them and cut flush with the end of the couple in order to present a shoulder for welding the heating element in place, as well as to

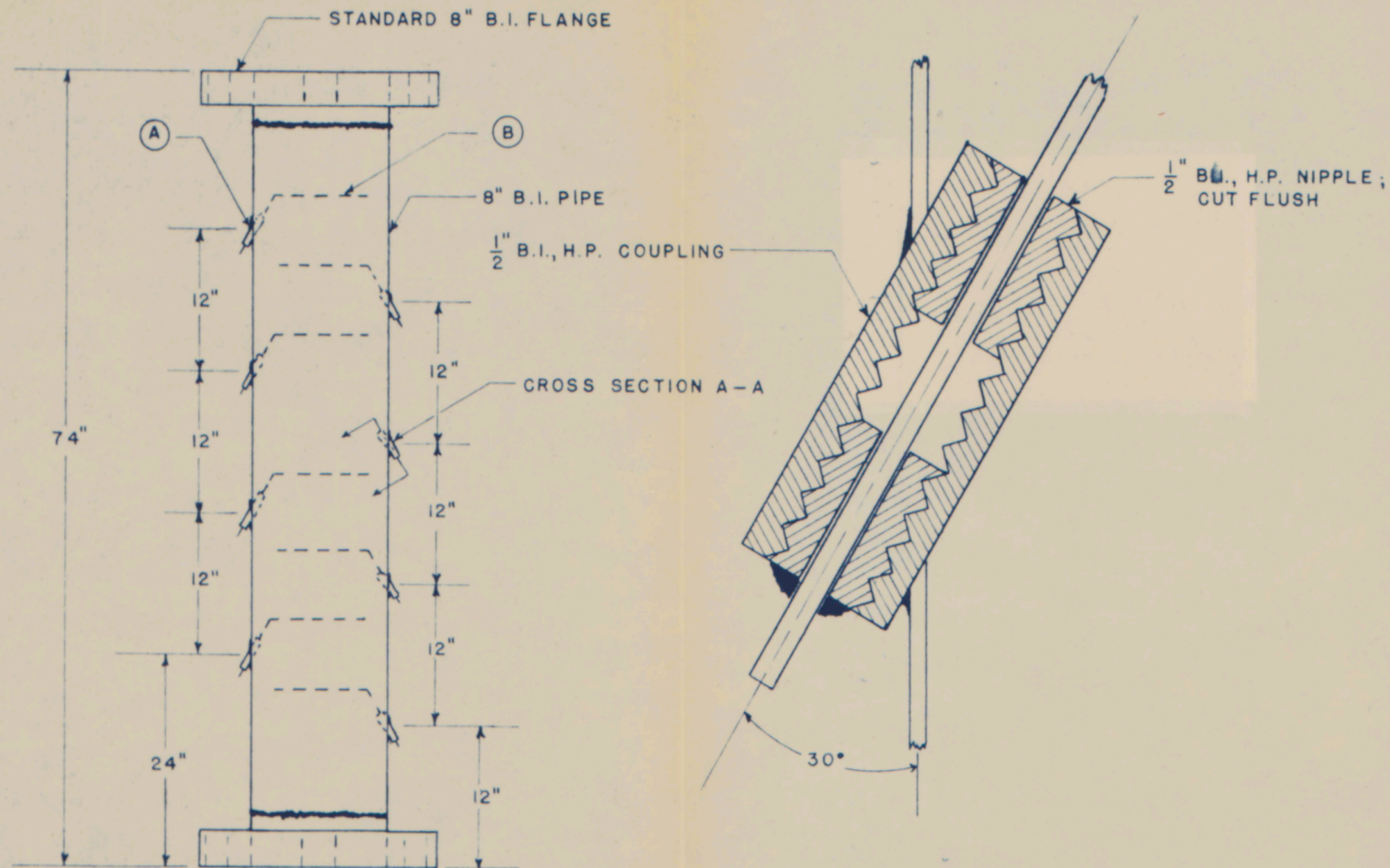
Figure 6
Heating Section

LEGEND

- (A) $\frac{1}{2}$ - IN. NIPPLE WELDED 30 DEGREE TO 8 - IN. PIPE WALL AND TANGENT TO A CIRCLE 6 - IN. DIAMETER FROM PIPE CENTER
- (B) HEATING ELEMENT, 6 - IN. IN DIAMETER, 1250 WATTS, 230/240 VOLTS

NOTES

- (1) HEATING ELEMENT LEAD OUT 0.505 - IN. IN DIAMETER
- (2) BLACK IRON-HIGH PRESSURE NIPPLES AND COUPLINGS USED
- (3) ALL WELDED CONSTRUCTION



ELEVATION

CROSS SECTION A-A

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HEATING SECTION

SCALE: NONE DATE: 2/20/55 CASE NO:
 DRAWN BY: J.G. Vignano FILE NO:
 CHECKED BY: J. E. McCall FIGURE NO: 8
 APPROVED BY: RWB 4/12/55 SHEET NO:

center the heating element lead-out shaft. The heating elements were of the cast-in-metal type, with the leads to the element cast in a shaft 0.505 inch in diameter and terminating from the element at an angle of 60 degrees from the horizontal and tangent to a circle of six inches. As each heater was inserted and welded in place, monel wire column packing was placed between the heating elements to increase the heating surface area. Attached to the bottom of the heating section, as shown in Figure 14, in appendix, was an 8-inch slip-on weld flange into which was welded a piece of 3/4-inch sheet steel with a 2-1/2-inch diameter hole in the center. A 2-1/2-inch diameter black iron pipe was welded in this hole. The 2-1/2-inch black iron pipe served as a gas transfer line to the fluidized retort, and as an auxiliary heating section. The 2-1/2-inch pipe was extended vertically downward from the heating section 28 inches, turned 90 degrees by means of a welded tube turn, and extended 24 inches horizontally, and then turned 90 degrees and extended 20 inches to the bottom of the flanged reducer on the bottom of the retorting section, as shown in Figure 14. The pipe was packed with monel wire column packing to increase the heating surface area. Strip heaters were attached to the transfer pipe to serve as auxiliary heaters and to reduce the time required to heat the gas to retorting temperature.

The entire unit and transfer pipe was insulated with 85 per cent magnesia.

Design and Construction of Cyclone Separator.

Because of the carry-over of solid particles from the disengaging section, with the shale oil vapors and fluidizing gases, it was necessary to install a cyclone separator to reduce the amount of solids passing into the condenser.

The first consideration in the design of the cyclone separator was the internal diameter required to effect separation of particles of 325-mesh (Tyler screen scale). This mesh was chosen as the highest concentration of small particles expected from the fluidized bed. Secondly, the gas velocities over the range as required for fluidization of the oil shale was considered as to the effect on the carry-over of solids. From the particle size range, gas velocity through the cyclone, and the pressure drop over the cyclone separator, it was calculated that the diameter of the separator should be one foot.

The cyclone separator was constructed from a one-foot diameter rolled sheet steel tank 48 inches long. The bottom of the tank was removed by means of a cutting torch, and a two-foot section of the tank removed from the remaining tank section. The bottom of the tank was then welded in place, with a 3/4-inch diameter black iron nipple 5 inches long welded in the center of the

bottom for removal of the solids. A tangential pipe $3/4$ inch in diameter and 8 inches long was welded to the top of the tank. A section of $3/4$ -inch black iron pipe, 9 inches long, was inserted in the center of the top of the tank so that the end inside of the tank was 3 inches below the incoming stream of gas from the tangential inlet. Angle iron strips were welded to the top of the assembled cyclone separator, Figure 9, page 80, to support it from the assembled frame.

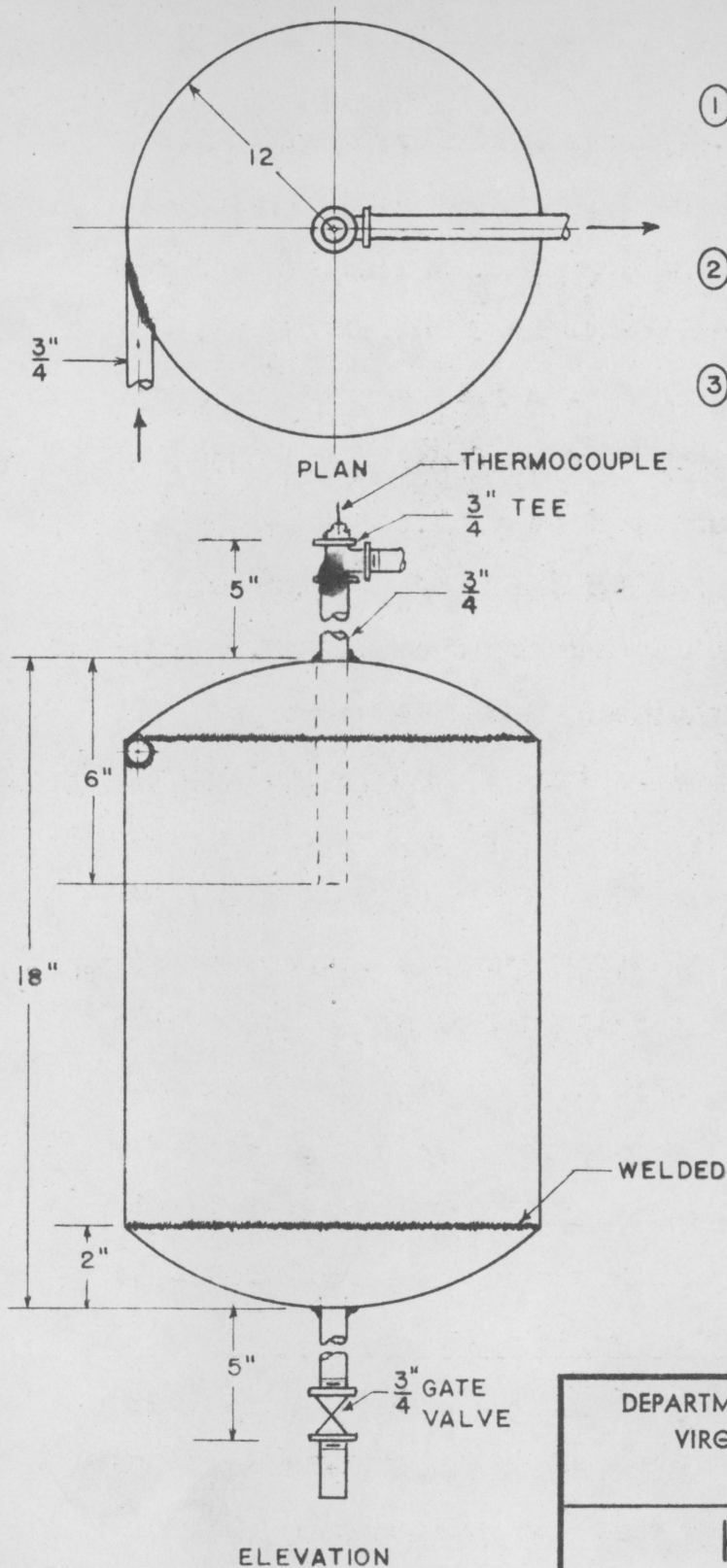
In order to prevent condensation of the oil vapor inside the cyclone separator, heaters were attached 8 inches below the top of the cyclone separator. Three 1000-watt heating elements were equally spaced around the separator, and were attached by means of $3/32$ -inch stud bolts welded to the tank wall. The heating elements were contained in ceramic plates.

The entire cyclone, except for the area occupied by the heating units, was insulated by the use of a two-inch layer of 85 per cent magnesia.

Design and Construction of Condensers. From previous experience it had been found that the heavier oil fractions blocked the condenser tubes by bridging over if the condenser temperature were below 100 °F. It was therefore decided to condense the shale oil vapors in fractions by designing the first condenser to operate at a minimum temperature of 250 °F, the

NOTES

- ① KNOCK-OUT TANK
CONSTRUCTED OUT OF $\frac{1}{4}$ -IN.,
ROLLED MILD STEEL
- ② HEATERS AND LAGGING
NOT SHOWN
- ③ BLACK IRON PIPING AND
FITTINGS USED



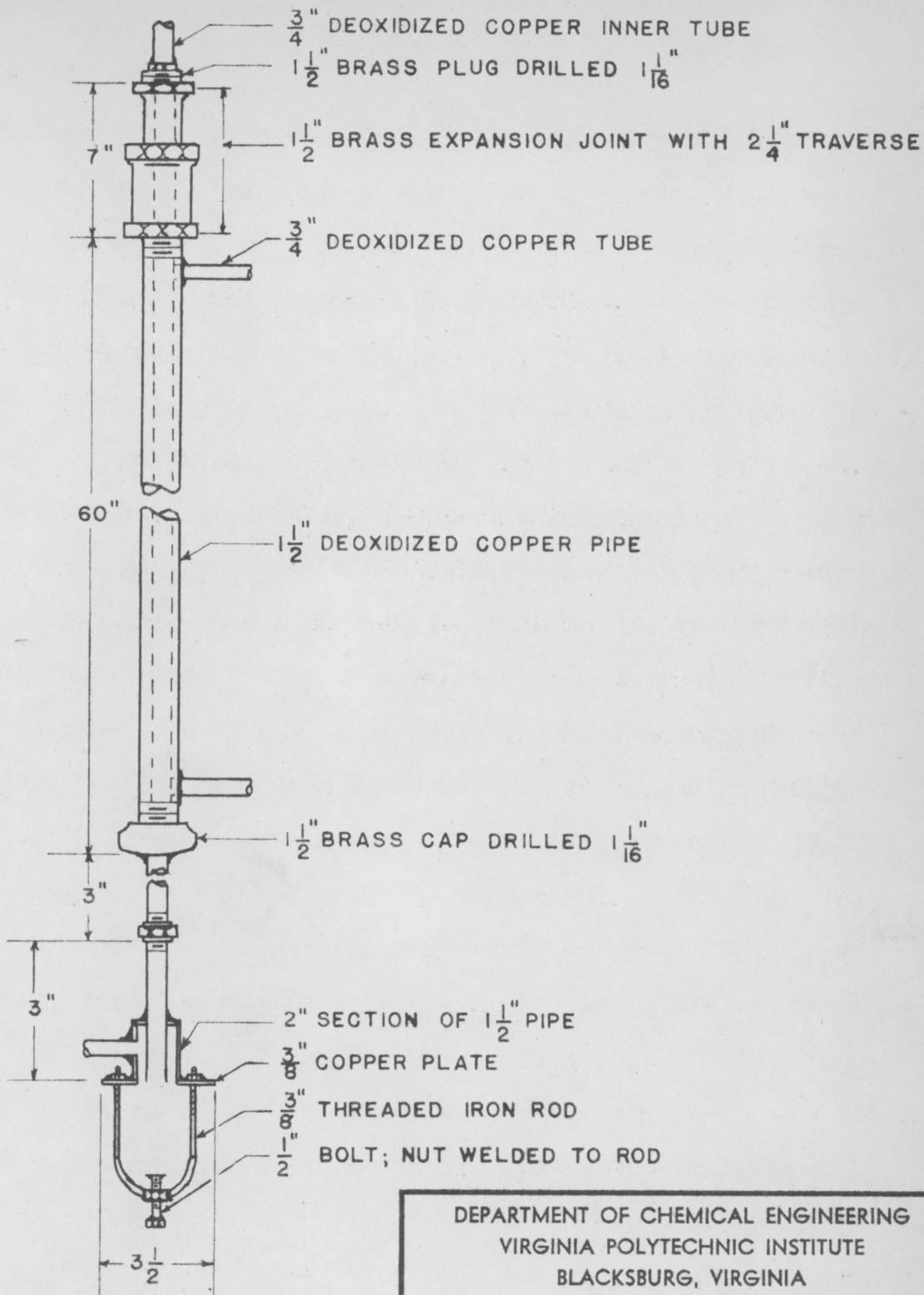
DEPARTMENT OF CHEMICAL ENGINEERING
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BLACKSBURG, VIRGINIA

KNOCK-OUT
TANK

SCALE: $\frac{3}{16}$ " = 1" DATE 2/4/55 CASE NO:
DRAWN BY: T.G.V FILE NO:
CHECKED BY: *James M. Carr, Jr.* FIGURE NO: 9
APPROVED BY: *F.W.B. 4/12/52* SHEET NO:

second condenser to operate at a minimum of 140 °F, the third condenser at a minimum of 60 °F, and the fourth condenser at the lowest possible temperature obtainable with melting ice in brine. At first only three condensers were designed, one to be brine cooled, one to be water cooled, and the third to be cooled by the gases returning from the brine cooled condenser. However, it was found after consideration of the maximum temperature of the entering gases to the gas-cooled condenser (600 °F), and the maximum temperature of the gases entering the water-cooled condenser (140 °F), the heat capacity of the gases, the heat capacity of the cooling medium, the heat transfer coefficient of the condenser tube, the film coefficient of the cooling medium and the material to be cooled, and the possible materials of construction, that the gas-cooled condenser was too long to be practical. It was therefore decided to construct the gas-cooled condenser as two separate units operating countercurrently as the first condenser.

From the above considerations and calculations it was indicated that the condenser tubes should be constructed from 3/4-inch deoxidized copper pipe, five feet long. As shown in Figure 10, page 82, the tube shell was constructed from 1-1/2-inch diameter deoxidized copper pipe, five feet long. The condenser tube was joined to the shell at the lower end by means of silver



ELEVATION

NOTES

- ① RECEIVER 2-IN. PYREX BOTTLE
- ② SILVER SOLDER USED IN WELDING

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SHALE OIL
 CONDENSER

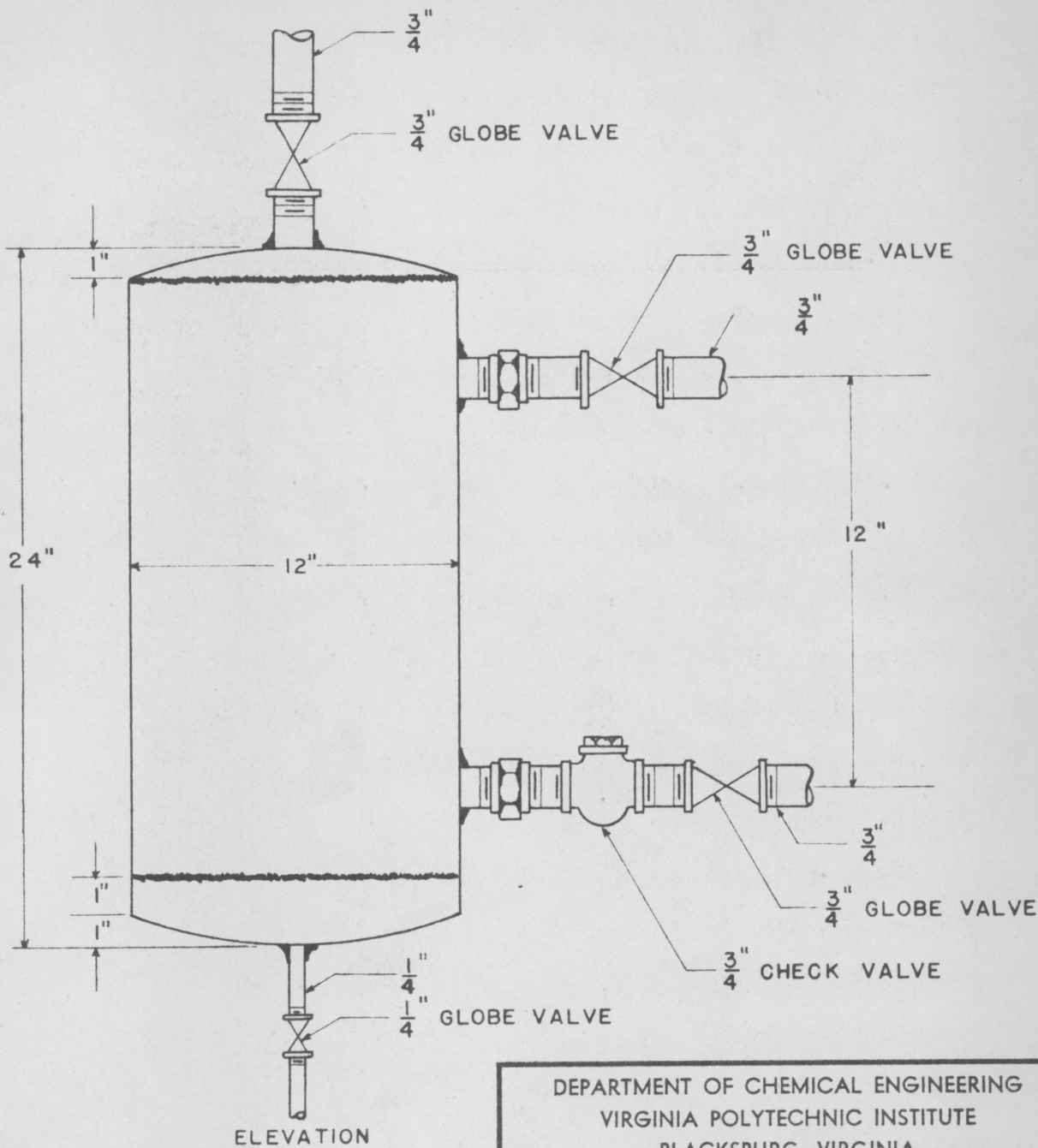
SCALE: NONE DATE 3/2/55 CASE NO:
 DRAWN BY: J.G. Vignano FILE NO:
 CHECKED BY: Jesse M. Carr, Jr. FIGURE NO: 10
 APPROVED BY: J.M.C. 4/12/55 SHEET NO:

soldering the tube into a brass 1-1/2-inch cap, and screwing the cap on the 1-1/2-inch pipe. At the upper end of the condenser a 1-1/2-inch expansion joint was screwed onto the shell of the condenser. A 1-1/2-inch brass plug which had been drilled 1-1/16 inches was screwed into the top of the expansion joint, and the 3/4-inch inner tube silver soldered to the brass plug. The inner condenser tube was joined to the oil receiver header by means of a 3/4-inch brass union. The oil receiver header, Figure 10, page 82, was constructed from a 3-inch section of 1-1/2-inch diameter deoxidized copper pipe with a 3-1/2-inch diameter copper flange on the bottom. A section of 3/4-inch deoxidized copper pipe extending through a 1-1/2-inch diameter copper flange was silver soldered to the top of the 3-inch section of pipe. A 3/4-inch outlet was provided above the inlet to reduce the amount of condensed oil carry-over to the next condenser. A receiver of pyrex glass was clamped to the bottom of the condenser. A silicone gasket was used to make a gas-tight seal between the condenser and pyrex receiver. A 3/4-inch inlet and outlet for the cooling medium was made in the condenser shell immediately above the cap on the shell and below the expansion joint at the top of the condenser.

All condensers were constructed in a similar manner. The condensers were insulated with 85 per cent magnesia. The assembly of the condensers into the retorting unit can be seen in Figure 14, found in the Appendix.

Design and Construction of Gas Surge Tank. In order to provide a reserve supply of fluidizing gas for the gas pump, a surge tank, 12-inch diameter by 24 inches long, was provided on the inlet side of the pumping section, as shown in Figure 11, page 85. A 3/4-inch diameter black iron couple was welded to the top of the tank, in the center, to provide an atmospheric blow-off line. On one side of the tank, five inches from the top, an inlet return-line connection was welded in place. Immediately below the inlet connection, an outlet connection to the pump was made by welding a 3/4-inch black iron couple to the tank wall. After welding the couples to the tank wall a hole, 7/8-inch in diameter, was drilled into the tank wall through the couple. A 1/4-inch black iron nipple was welded to the bottom of the tank to provide a blow-down line to the sewer for any undesirable material which collected in the surge tank.

The assembly of the surge tank into the fluidisation unit is shown in Figure 14, in the Appendix.



NOTES

- ① 1/4 - IN. MILD STEEL USED IN CONSTRUCTION OF TANK
- ② BLACK IRON PIPING USED

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SURGE TANK

SCALE: NONE DATE 3/1/55 CASE NO:
DRAWN BY: J. G. Vignano FILE NO:
CHECKED BY: Jesse M. Carr Jr. FIGURE NO: 11
APPROVED BY: FWB 4/12/55 SHEET NO:

Design and Construction of Gas-pumping Section.

A gas pump was provided for moving the fluidizing gas through the closed system. The pump was a sliding-vane type which was internally lubricated and had a capacity of 20 cubic feet per minute at atmospheric pressure. The pump was designed to operate at 300 revolutions per minute, and was driven by a 1-1/2 horsepower three phase motor, through a three-inch diameter pulley on the motor to a 24.8-inch pulley on the pump. The pulleys were designed for "A" size belts.

In order to prevent carry-over of the oil from the pump to the condensers, an oil filter was placed on the outlet of the pump. The oil filter was constructed from an 18-inch section of 3-inch black iron pipe which was packed with monel wire column packing and capped on both ends by means of 1/4-inch steel plate welded to the pipe. A 3/4-inch nominal pipe size hole was drilled and tapped into the top steel plate as an outlet for the pumped gases, and in the bottom steel plate a 1-inch pipe size hole was drilled and tapped for the inlet gases from the pump. A pressure tap was made into the side of the filter two inches below the top steel plate by welding a 1/4-inch black

iron couple to the pipe wall, and then drilling through the pipe wall.

A 1/4-inch couple was welded to the side of the filter immediately above the bottom plate for cleaning the entrained oil from the filter. The oil was returned to the pump on the low pressure side by means of a 1/4-inch copper tube. This arrangement provided for continuous sealing and lubrication of the pump. The inlet gas line from the pump to the filter extended two inches inside the filter to provide a reservoir for entrained lubricating oil. The gas-pumping section was mounted one foot from the floor on an angle iron and one-inch black iron pipe.

In order to control the flow of gas from the pump through the fluidized unit, a 1/2-inch by-pass line was provided, with a 1/2-inch brass globe valve for control. The by-pass was situated immediately above the oil filter and connected the high pressure side of the pump to the low pressure side. The gas-heating section was connected to the pumping section on the high pressure side through a 3/4-inch brass gate valve and a run of 3/4-inch black iron pipe.

Design and Construction of Orifice. From the estimated gas volume to be pumped through the fluidization system, a thin plate orifice was designed for the return line from the annular space of the first condenser

to the surge tank. The return line was constructed from 3/4-inch diameter black iron pipe. The orifice plate was gasketed between a pair of 3/4-inch flanges and orifice taps were made eight pipe diameters from the face of the orifice upstream, and 1-1/2 pipe diameters downstream from the orifice. The orifice taps were made by welding 1/4-inch black iron couples to the pipe and drilling a 1/8-inch hole through the pipe wall inside the couple welded on the pipe. The pressure differential was transferred through 1/8-inch copper tubing to the manometers mounted on the panel board.

Design and Construction of Panel Board, Control and Measuring Units. The panel board was constructed from 5/8-inch 4-ply plywood. The panel board was designed six feet long and four feet wide. The main electrical supply of 440 volts was brought through a 90 ampere fused switch. From this switch, as shown in Figure 12 of the Appendix, 440 volts without a ground was wired to the pump switch, and 220 volts with ground was wired into the distribution box with magnetic breakers. The motor was wired directly from the motor switch, and all wiring from the distribution box was connected through separate switches with pilot lights. The strip heaters on the gas transfer pipe from

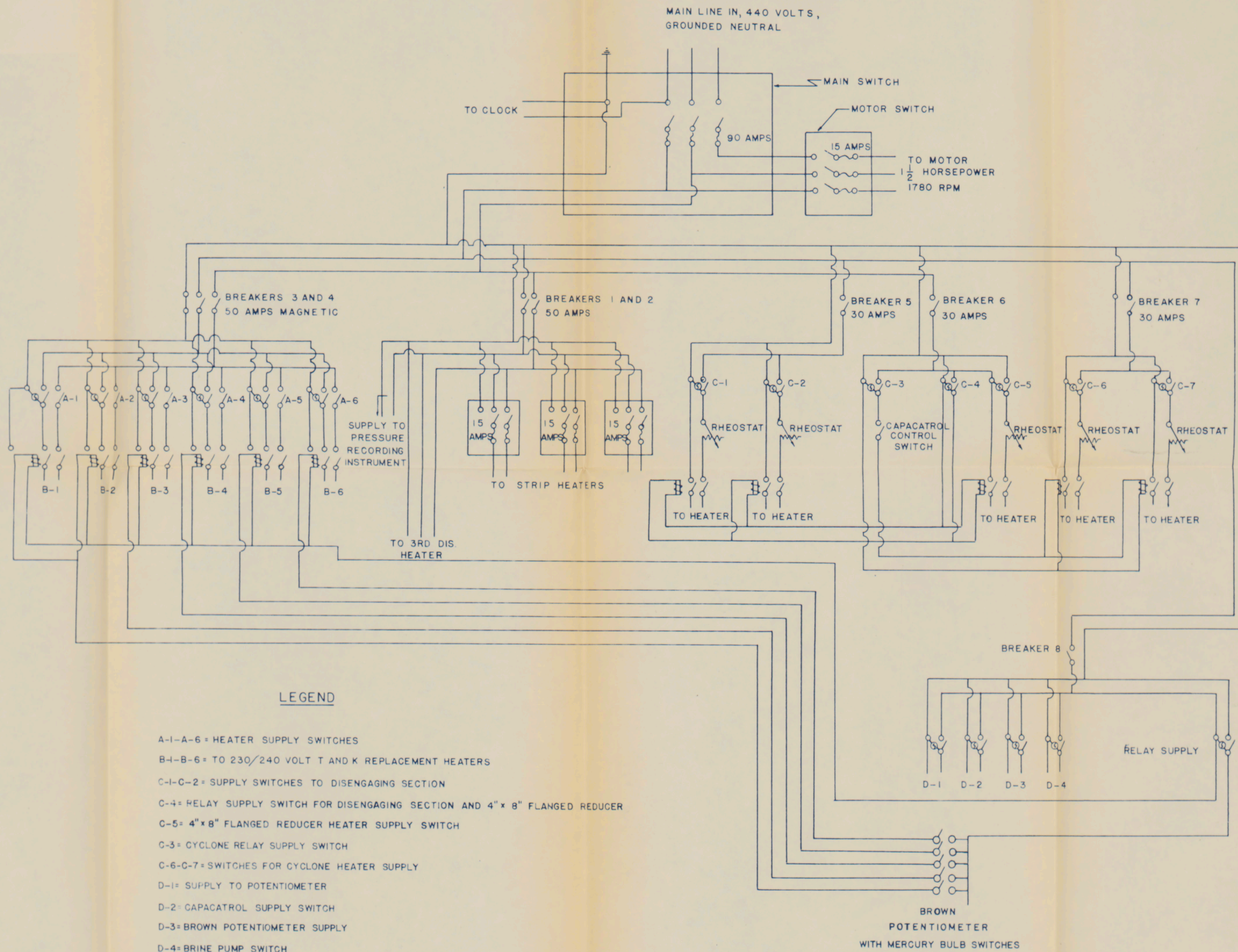
Figure 12

Wiring Diagram for Fluidized Retorting Unit

(see pocket on back cover)

Figure 12

Wiring Diagram for Fluidized Retorting Unit



LEGEND

- A-1-A-6 = HEATER SUPPLY SWITCHES
- B-1-B-6 = TO 230/240 VOLT T AND K REPLACEMENT HEATERS
- C-1-C-2 = SUPPLY SWITCHES TO DISENGAGING SECTION
- C-4 = RELAY SUPPLY SWITCH FOR DISENGAGING SECTION AND 4" x 8" FLANGED REDUCER
- C-5 = 4" x 8" FLANGED REDUCER HEATER SUPPLY SWITCH
- C-3 = CYCLONE RELAY SUPPLY SWITCH
- C-6-C-7 = SWITCHES FOR CYCLONE HEATER SUPPLY
- D-1 = SUPPLY TO POTENTIOMETER
- D-2 = CAPACATROL SUPPLY SWITCH
- D-3 = BROWN POTENTIOMETER SUPPLY
- D-4 = BRINE PUMP SWITCH

NOTES

- ① ALL BREAKERS ARE MAGNETIC TYPE IN ONE DISTRIBUTION PANEL
- ② SWITCHES WITH INDICATING BULB ARE MANUAL SWITCHES FOR OPERATION
- ③ RELAYS, POTTER AND BRUMFIELD TYPE, PR SERIES 7A DOUBLE BREAK, NORMAL OPEN, RATED AT 20AMPS WITH SILVER CONTACTS, COIL VOLTAGE 110 VOLTS A.C.

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WIRING DIAGRAM FOR
 FLUIDIZED RETORTING UNIT

SCALE: NONE DATE: 1/11/55 CASE NO.
 DRAWN BY: J.G. Vignone FILE NO.
 CHECKED BY: J. M. ... FIGURE NO. 12
 APPROVED BY: JMB 4/12/55 SHEET NO.

the gas-heating section to the retort were controlled by three knife-edge switches which controlled two banks of three heaters and one bank of six heaters.

The air-heating section was controlled by a thermocouple in the bottom of the fluidized bed, actuating a controller, which by means of mercury switches, controlled the coil power of relays which carried power for the heating elements. As the temperature of the retort approached the retorting temperature, the mechanism of the controller turned off the power to the heaters in multiples of one. Thus, some degree of proportional control was accomplished.

For control purposes, thermocouples were placed in all entering and exit streams, as indicated in Table VI, page 91. The thermocouples were constructed from 1/8-inch diameter iron tube with an asbestos insulated constantan wire passing through the tube, with one end of the wire welded to the iron tube by use of a carbon arc. The welding of constantan wire to the iron tube closed the tube. The thermocouple thus constructed was inserted through a 1/8-inch ferrule-type copper tube to pipe adapter. The ferrule-fitting was then screwed into a 1/8-inch pipe fitting in the piece of equipment in which the temperature was to be measured. Thermocouple leads of separate strands of iron and constantan wire, enameled with a single cotton cover, were

TABLE VI

Positions of Thermocouples in Fluidized Retorting Unit

Thermocouple No	Position in Retorting Equipment
1	Top of fluidized bed
2	Middle of fluidized bed
3	Disengaging section
4	Gas into top of first condenser
5	Gas out of the bottom of first condenser
6	Gas into the top of the second condenser
7	Gas out of the bottom of the second condenser
8	Gas into the top of the third condenser
9	Gas out of the bottom of the third condenser
10	Gas into the top of fourth condenser
11	Gas out of the bottom of the fourth condenser
12	Brine into the bottom of the fourth condenser
13	Brine out of the top of the fourth condenser
14	Water into the bottom of the third condenser
15	Water out of the annular space of third condenser
16	Gas out of top of annular space of second condenser
17	Gas into the annular space of first condenser
18	Gas out of the annular space of the first condenser to return line for surge tank
Capacitrol	Cyclone separator
Temperature Controller	Immediately above the distribution plate in fluidized bed

welded to the iron tube and constantan wire of the thermocouple respectively. The leads were wired into the indicating potentiometer. The indicating potentiometer was mounted on the panel board immediately above the recording desk.

The manometers required for measuring gas flow and the pressure drop across the fluidized bed were mounted on the panel board.

For control of the heating elements on the disengaging section, rheostats were placed on the positive side of the lead wire from the switches to the heaters.

Control of the temperature of the cyclone separator was accomplished by a control instrument operating from a thermocouple in the exit gas line of the cyclone separator. The control instrument operated relays carrying the power load of the heaters. Also, rheostats were used to control the temperature of operation of the heating elements.

In Figure 13, page 93, the lay-out of the panel board is shown.

Design and Construction of Fluidization Unit Supports.

All sections of the fluidization unit were supported on a frame constructed from 1-1/4-inch black iron pipe. Pipe sections 10-1/2-feet long were screwed into floor flanges attached to the floor by means of bolts passing through the



Figure 13. Panel Board of Fluidized Retorting Unit

flanges into korkers driven into holes drilled in the floor. The tops of the pipes were held together by welding each individual pipe to a section of 1-1/2-inch pipe running the length of the unit. Cross-members were then welded to the horizontal pipes to make the unit rigid. The heating section, fluidization section, cyclone, and condensers were supported from the top of the framework. The gas pump and motor, and surge tank, were supported from lower sections of the frame.

To simplify the wiring of thermocouples and heating units, two 1 x 8-inch boards were attached to the back of the supports six feet off the floor. The board afforded an easy method of supporting and tracing all the wiring since each wire was individually stapled to the boards.

Calibration of Thermocouples. All thermocouples were wired completely and then calibrated at the melting point of ice and at known water temperatures. The indicating instrument was readable, by interpolation, to 1 °F, however the instrument was calibrated in 5 °F intervals.

Calibration of Orifice. The orifice for measuring gas flow through the system was calibrated by means of an air flow meter. The air flow meter was placed on the exit side of the orifice and the calibration readings were taken at various pressure drops across the orifice, as indicated on the manometer. The reading of the air flow meter was plotted against the pressure drop across the orifice and is shown in Figure 16, page

Assembly of Fluidization Unit. The fluidization unit was assembled as shown in Figure 14 in the Appendix. All units were assembled in such a way that they could be removed from the assembled unit without removing or disturbing other units.

Observation of Fluidization Characteristics of Oil Shale. Before retorting tests were made, oil shale of various mesh sizes was fluidized in a four-inch glass column, and the gas volume required for fluidization determined. During fluidization in the glass column, the characteristics of the oil shale in the fluidized bed were observed.

Preparation of Oil Shale for Retorting. The oil shale as received, in 1/2-inch size lumps, was crushed in a jaw-crusher. The material from the jaw-crusher was screened through a bank of screens. The screens were arranged from top to bottom according to increasing mesh size, 16, 28, 70, 100, 200, and pan (Tyler screen scale). A representative sample, one-fourth pound of crushed oil shale, was placed on the top screen and the screens shaken mechanically for seven minutes. The screens were then disassembled and their contents placed in separate labeled, sealed, one-gallon containers, except for the oil shale on the top, 16-mesh screen. The material on the 16-mesh screen was placed in a container and later passed through the jaw-crusher again and re-screened.

Figure 14

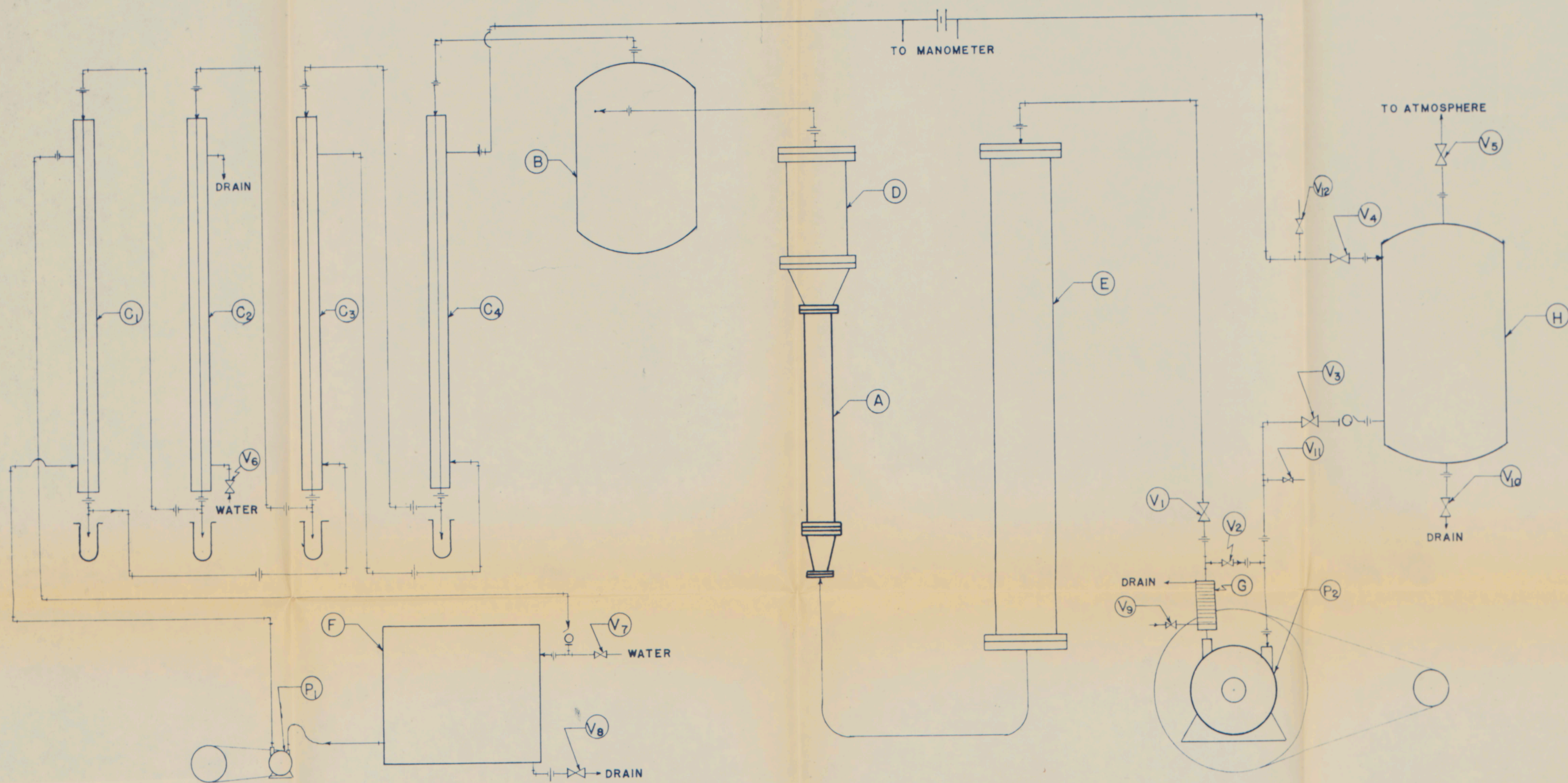
Schematic Diagram of Retorting Unit

(see pocket on back cover)

14

Figure 14

Schematic Diagram of Retorting Unit



LEGEND

- (A) RETORT
- (B) KNOCK-OUT TANK
- (C₁ - C₄) CONDENSERS
- (D) DISENGAGING SECTION
- (E) HEATING COLUMN
- (F) BRINE TANK
- (G) OIL SEPARATOR
- (H) SURGE TANK
- (P₁ - P₂) PUMPS
- (V₁ - V₁₂) CONTROL VALVES

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SCHEMATIC DIAGRAM OF RETORT UNIT		
SCALE: NONE	DATE: 4/1/55	CASE NO:
DRAWN BY: J. B. ...	FILE NO:	
CHECKED BY: J. B. ...	FIGURE NO: 14	
APPROVED BY: J. B. ...	SHEET NO:	

The jaw-crusher was used to prepare the oil-shale required for tests using minus 16 plus 28, minus 28 plus 70, and minus 70 plus 100 mesh (Tyler screen scale).

The amount of oil shale needed for test number four, minus 100 plus 200 mesh, could not be obtained from the jaw-crusher. The oil shale remaining on the 16-mesh screen was placed in a rod mill with 20-pounds of rods, and ground for 5 hours. The material was then screened and oil shale collected for test number four.

The material for each test was quartered and sampled for the determination of shale oil content. The sample for analysis was stored in a sealed pint jar. The remaining oil shale was stored in labeled, sealed, one-gallon containers until used for retorting.

Operation of Fluidization Unit. The procedure followed in operation of the fluidized retort is outlined in the following paragraphs, and all valve numbers are in reference to Figure 14 in the Appendix.

Charging of the Retort. Sixteen and one-quarter pounds of oil shale of a known particle size was placed in the retorting section, which had been removed from the system. The retort was then placed in the system, gasket placed between the flanges, the flange bolts tightened, the thermocouples inserted into the bed and secured, and the manometer leads attached to the retort.

Coolant for Condensers and Cooling Coil. Before applying heat to the system, ice was placed in the brine tank and valve V-8 closed. Salt was placed over the ice and a brine solution made in the tank, and if needed, tap water was added by opening valve V-7. Additional ice was added during the retorting test as needed. The main switch was closed and all magnetic breakers closed. The brine pump was then started and the brine allowed to circulate. Valve V-6 was opened, allowing the cooling water to circulate through the condenser. Valve V-9 for cooling water to the oil separator, was opened.

Determining Fluidization Gas Velocity. Before beginning heating of the retorting unit, the gas flow rate through the unit was determined for maximum fluidization of oil shale by the following procedure. Valves V-1, V-10, and V-12 were closed, and valves V-2, V-3, V-4, V-5, and V-11 were opened. The gas pump was then started, and V-4 partially closed. Valve V-1 was then slowly opened at the same time observing the pressure drop across the fluidized bed. If fluidization was not accomplished by opening V-1, then valve V-2 was gradually closed until the pressure drop across the fluidized bed indicated slugging fluidization. The valve V-2 was then opened slightly until normal fluidization was indicated by the pressure drop across the

fluidized bed. Valve V-4 was then closed completely and the system pressurized to ten pounds per square inch gage, as indicated on the pressure recorder. At this time valve V-5 was closed and valve V-4 was opened very slowly, until the valve was fully open. The pressure drop across the fluidized bed was observed during the opening of the valve, and if slugging flow was indicated, valve V-2 was opened slightly to return the bed to normal fluidization. When valve V-4 was opened completely and the equipment operating normally, the gas pumped was stopped. The air in the system was then bled off carefully through valve V-5 until the pressure in the system was atmospheric.

Purging of Fluidization Unit. To prevent combustion of oil shale during retorting, the fluidization unit was purged with an inert gas, carbon dioxide. The following procedure was used. Valve V-3 was closed and V-5 opened. Carbon dioxide was then bled into the system through valve V-11, and purging continued for five minutes. The system was then pressurized with carbon dioxide to ten pounds pressure by closing valve V-5. Valve V-11 was closed and valve V-3 opened, and the gas pump started. After circulation of the gas for ten minutes, valve V-12 was opened and a sample of the gas taken. The gas sample was analyzed for oxygen content. If the oxygen content

were below one per cent, the system was considered ready for operation. When the oxygen content was found to be above one per cent, the pressure in the system was carefully bled down to atmospheric through valve V-5. The system was then again pressurized as above and another gas sample analyzed. In any event the purging procedure was continued until the oxygen in the gas was one per cent or less.

Operation of Retorting Unit. After purging was completed and the system was pressurized to ten pounds per square inch gage, all heater switches were closed as well as all the accessory switches, except for the supply switch to the heater control instrument switch. The range of the mechanical controller prohibited its proper operation until the temperature of the retort reached 250 °F. During the heating period, a record of the temperature over the fluidisation unit was made every fifteen minutes. A record of the gas flow rate and the pressure drop over the fluidized bed was also made.

When the retort reached a temperature of 250 °F, the mechanical controller electrical supply was turned on, and as the temperature of the system increased, additional back electromotive force was added to the thermocouple circuit to extend the range of the instrument

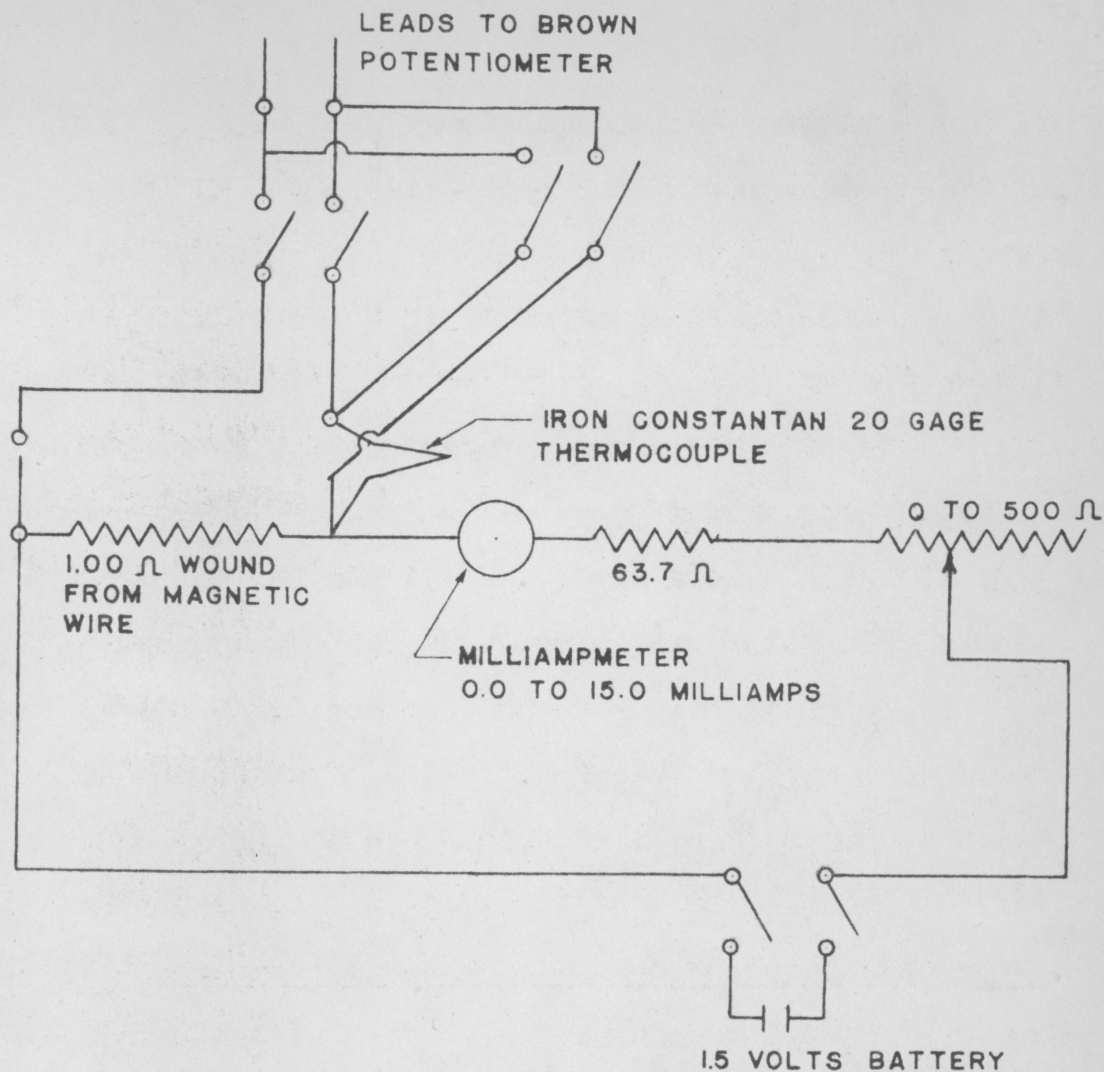
to 715 °F. The circuit for adding the electromotive force in a negative direction is shown in Figure 15, page 102.

After reaching the retorting temperature of 715 °F, the mechanical controller controlled the temperature of the inlet gas to the retort. It was found that the controller controlled the temperature of the gas to the retort to within ± 5 °F. Retorting of the oil shale was continued for one hour, after which all heaters were turned off. The gas pump was also stopped and a sample of the retort gas taken. The gas of the system was then bled off through a gas meter to determine the volume in the system.

Determining the Gas Generated During Retorting.

From the volume of the gas, pressure, and the amount of the gas added to the system, as well as the volume and temperature of the fluidization system, the volume of the gas generated in the retort during retorting was determined.

Recovery of Shale Oil. After retorting, the condensers were allowed to drain for 24 hours and then washed with ethyl ether. The material obtained was filtered in a soxhlet extraction tube to remove any trace of solids in the material. The extraction thimble was then extracted with ether until the liquid from the tube was clear. The ether was then removed from the shale oil by distillation.



NOTE

- ① ALL SWITCHES DOUBLE POLE, SINGLE THROW SWITCHES

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RANGE EXTENDER FOR
BROWN MECHANICAL
POTENTIOMETER

SCALE: NONE DATE 4/4/55 CASE NO:
DRAWN BY: T.G. Viganò FILE NO:
CHECKED BY: *John M. Carr, Jr.* FIGURE NO: 15
APPROVED BY: *JMC 4/12/55* SHEET NO:

Analysis of Retort Gas. After retorting, the gas from the retort was sampled and the carbon dioxide, oxygen, carbon monoxide, hydrogen, hydrogen sulfide, nitrogen, and non-condensable hydrocarbon gases were determined. The analysis of the retort gas was determined by use of an orsat gas analysis apparatus.

Oil Shale Analysis. Samples of the oil shale before and after retorting were analyzed for shale oil content by use of a modified Fischer retort. The shale oil content was reported in weight per cent.

Determination of the Specific Gravity of Shale Oil. The specific gravity of shale oil was determined according to American Society of Testing Materials method D 70-27^(107a). The determination was made at 60 °F in a constant-temperature bath.

Determination of Viscosity of Shale Oil. The kinematic viscosity of shale oil was determined according to American Society for Testing Materials method D 445-46 T^(108a). The viscosity determinations were made at 210 °F in a constant-temperature bath.

Determination of ASTM Distillation Mid-boiling Point. The distillation of the shale oil samples was performed according to the procedure described by the American Society for Testing Materials method D 285-41^(106a).

Determination of Characterisation Factor. In an effort to compare the shale oil samples on a basis common to the oil industry, the characterization factor was determined. The characterization factor of the shale oil samples was determined. From the distillation of the shale oil, the mid-boiling point of the shale oil samples was determined. The characterization factor of the shale oil samples was determined by plotting the degrees A.P.I. against the kinematic viscosity of the shale oil samples, Figure 17, page 120.

Data and Results

The following paragraphs contain the data and results of retorting tests, analysis data of oil shale and shale oil, and calibration data obtained for the orifice used to measure the gas flow through the retorting system.

Calibration Data of Orifice. The orifice calibration data is plotted on Figure 16, page 107, in cubic feet per minute versus pressure drop in inches of mercury. Table VII contains data used in plotting the calibration curve.

Summary of Data and Results. A summary of the data and results of retorting oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale) for one hour in a 4.026-inch diameter fluidized bed, using carbon dioxide at 715 °F as the fluidizing medium, is contained in Table VIII, page 108. Data and results obtained from the analysis of the oil shale, shale oil, and retort are also included in Table VIII.

TABLE VII

Calibration Data for Thin Plate Orifice Used for
Measuring Gas Flow in Oil Shale Retorting Unit

Reading No	Manometer Reading In. Dibutyl Phthalate	Meter Reading cu ft/min
1	0.05	1.0
2	0.25	2.0
3	0.45	3.0
4	0.70	4.0
5	1.00	5.0
6	1.50	6.0
7	2.00	7.0
8	2.75	8.0
9	3.30	9.0
10	4.0	10.0
11	4.9	11.0
12	5.9	12.0
13	6.95	13.0
14	8.2	14.0
15	10.0	15.0

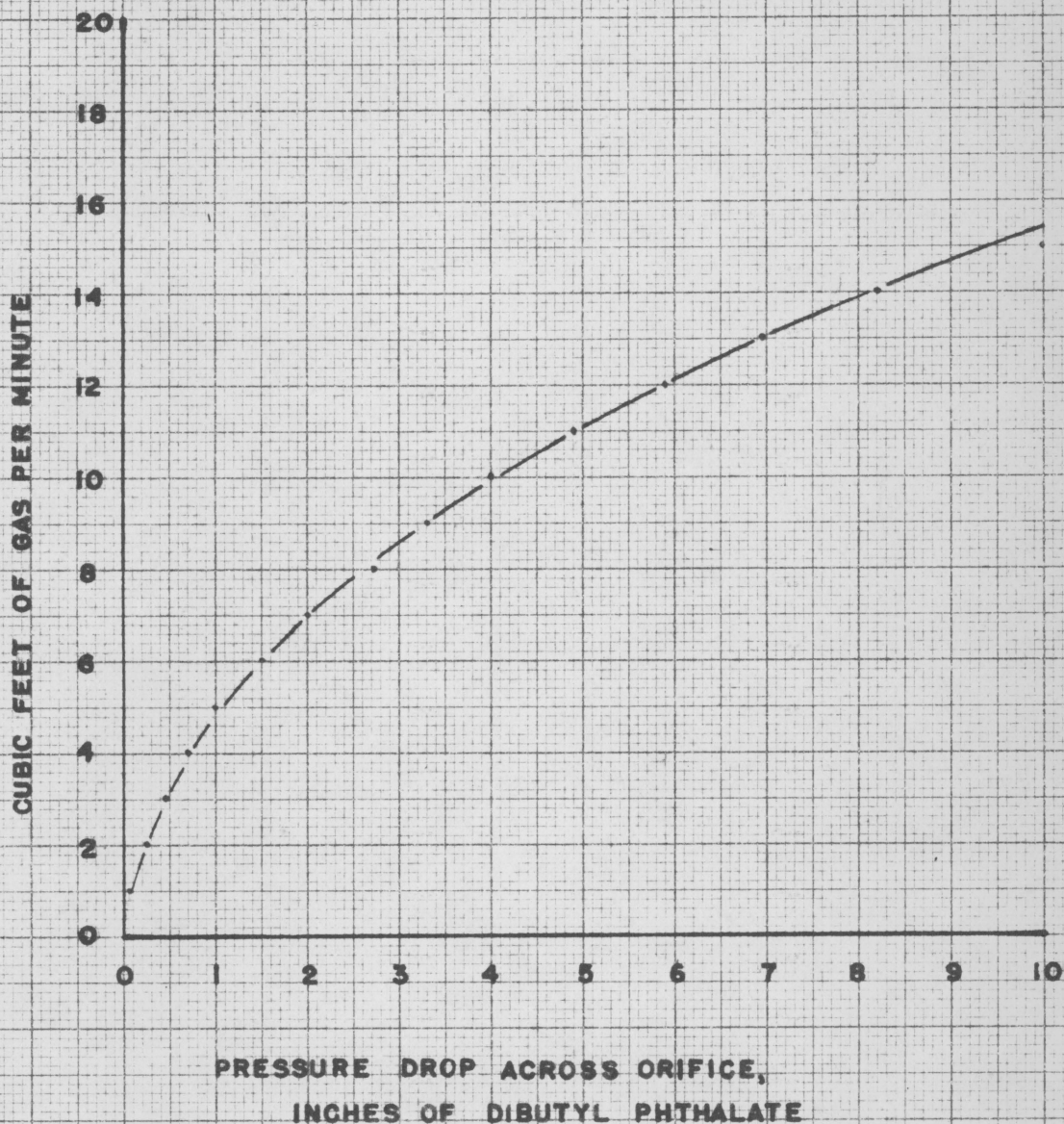


FIGURE 16. CALIBRATION CURVE FOR THIN PLATE ORIFICE USED FOR MEASURING GAS FLOW IN OIL SHALE FLUIDIZED RETORTING UNIT

Table VIII

Summary of Data and Results of the Fluidized
Retorting of Oil Shale

(see pocket on back cover)

Table VIII

Summary of Data and Results of the Fluidized
Retorting of Oil Shale

TABLE VIII

SUMMARY OF DATA AND RESULTS OF THE FLUIDIZED RETORTING OF OIL SHALE

RETORTING TEST NO	CHARGE PARTICLE SIZE MESH	CHARGE WEIGHT LBS	RETORTING TEMPERATURE °F	RETORTING TIME HR	RETORTING PRESSURE LBS, GAGE	^a OIL SHALE DATA			ANALYSIS OF SHALE OIL					RETORTING GAS VELOCITY CU FT PER MIN	VOLUME OF GAS GENERATED CU FT	^b GAS ANALYSIS						
						RAW SHALE % OIL	RETORTED SHALE % OIL	OIL YIELD %	A.P.I. GRAVITY DEGREES	VISCOSITY AT 210 F CENTIPOISES	MID-BOILING POINT °F	AVERAGE BOILING POINT °F	CHARACTERIZATION FACTOR			CO ₂ %	O ₂ %	CO %	N %	H ₂ S %	H ₂ %	ETHANE %
1	-16 +28	16.25	715	1	10	17.3	8.6	50.5	15.2	12.18	510	824	11.2	14.6	0.67	84.8	0	4.7	0	1.2	0.6	9.0
2	-28 +70	16.25	715	1	10	17.1	9.0	47.3	17.9	4.64	500	730	11.4	10.5	0.72	84.1	0	8.4	0	1.3	0.6	12.0
3	-70 +100	16.25	715	1	10	15.4	9.1	40.9	18.6	2.94	482	690	11.2	7.0	1.01	83.0	0	0.6	0	0.8	1.4	14.6
4	-100 +200	16.25	715	1	10	15.0	10.6	39.4	22.4	1.66	380	590	11.0	5.0	1.20	62.1	0	4.3	0	1.0	10.2	22.3

^a OIL SHALE DATA IN WEIGHT PER CENT^b GAS ANALYSIS REPORTED IN VOLUME PER CENT

Sample Calculations

The sample calculations include calculations for the design of the gas-heating unit, the condensers, the determination of shale oil yield, specific gravity, A.P.I. gravity, kinematic viscosity, and determination of characterization factor.

Design Calculations for Gas-heating Section. Calculations of the gas flow rate, and the number of heating elements required to heat the gas to 1000 °F are given in the following paragraphs. All heating calculations are based on six-inch diameter 1250-watt heaters.

Calculation of Gas Flow Rate through Gas-heating Section. To accommodate the six-inch diameter heating elements, the diameter of the gas-heating was made eight inches. The mass gas flow rate was based on the maximum available from the gas pump, 20 cubic feet per minute.

$$w' = V_g a \quad (1)$$

where

w' = air flow rate, lb/min

V_g = air flow rate at standard conditions,
cu ft/min

then

$$w' = \frac{\text{cu ft}}{\text{min}} \times \frac{\text{lb}}{\text{cu ft}} = \text{lb/min}$$

$$w' = 20 \times 0.0808$$

$$w' = 1.616 \text{ lb/min.}$$

The following is the calculation of the mass superficial air velocity of the gas through the gas-heating section:

$$G = \frac{W}{A_t} \quad (2)$$

where

G = mass superficial gas velocity,
lb/hr-ft²

W = gas velocity, lb/hr

A_t = area of heating section, ft²

then

$$G = \frac{1b}{hr} \times \frac{1}{ft^2} = 1b/hr-ft^2$$

$$G = \frac{1.616 \times 60}{0.347}$$

$$G = 28 \text{ lb/hr-ft}^2.$$

Calculation of Heater Requirement. Based on a maximum mass superficial gas velocity of 28 pounds per hour-square foot of pipe cross-section and a required temperature of the gas of 1000 °F, the following calculation was made:

$$q = w C_p \Delta T \quad (3)$$

where

q = heat requirement, Btu/hr

w = air flow rate, lb/hr

C_p = heat capacity of gas, Btu/lb-°F

ΔT = temperature differential between inlet
and outlet gas, °F

then

$$q = \frac{\text{lb}}{\text{hr}} \times \frac{\text{Btu}}{\text{lb-°F}} \times \text{°F} =$$

$$q = 96.96 \times 0.253 \times 920$$

$$q = 2695 \text{ Btu/hr.}$$

Determination of the Number of Heating Units Required. Based on 1250 watts per heating unit and a requirement of 2695 Btu per hour to heat the gas, the number of heating elements was calculated from:

$$N = \frac{q}{E \times H} \quad (4)$$

where

N = the number of heating units required,
no units

q = heating requirement to heat the gas to
1000 °F, Btu/hr

E = watts per hour per Btu, 0.293 Btu/watt-hr

H = watts per heating unit, 1250 watts/per unit

then

$$N = \frac{\text{Btu}}{\text{hr}} \times \frac{\text{watt-hr}}{\text{Btu}} \times \frac{\text{units}}{\text{watt}} = \text{units}$$

$$N = \frac{2695}{0.293 \times 1250}$$

$$N = 7 \text{ units.}$$

Design Calculations for First Gas-cooled Condenser.

Based on a gas velocity of 15 pounds per hour, a hot gas inlet temperature of 600 °F, a hot gas outlet of 250 °F, and a coolant inlet temperature of 40 °F, the following calculations were made. The amount of heat lost by the hot gas was:

$$q = w C_p \Delta t \quad (5)$$

where

q = heat lost, Btu/hr

w = air flow rate, lb/hr

C_p = specific heat of the gas, Btu/lb-°F

Δt = temperature differential between inlet and outlet gas, °F

then

$$q = \frac{\text{lb}}{\text{hr}} \times \frac{\text{Btu}}{\text{lb-}^\circ\text{F}} \times ^\circ\text{F} = \text{Btu/hr}$$

$$q = 15 \times 0.30 \times (600 - 250)$$

$$q = 1575 \text{ Btu/hr.}$$

The temperature of the outlet cooling gas was determined from

$$T = \frac{q}{C_p \times w} + t_1 \quad (6)$$

where

T = temperature of exit cooling gas, °F

q = heat lost by gas being cooled, Btu/hr

w = coolant flow rate, lb/hr

C_p = specific heat of coolant, Btu/lb-°F

t₁ = inlet temperature of the coolant, °F

then

$$T = \frac{\text{Btu}}{\text{hr}} \times \frac{\text{hr}}{\text{lb}} \times \frac{\text{lb-}^\circ\text{F}}{\text{Btu}} + ^\circ\text{F} = ^\circ\text{F}$$

$$T = \frac{1575}{1.0 \times 15} + 40$$

$$T = 145 \text{ } ^\circ\text{F.}$$

For use in determining the condenser surface area required to cool the gas the logarithmic mean temperature difference was determined from:

$$\Delta t_m = \frac{\Delta t_1 - \Delta t_2}{\ln \frac{\Delta t_1}{\Delta t_2}} \quad (7)$$

where

Δt_m = logarithmic mean temperature difference, °F

Δt_1 = temperature difference entering hot gas and leaving cooling gas, °F

Δt_2 = temperature difference of the leaving hot gas and the entering cooling gas, °F

then

$$\Delta t_m = \frac{(°F - °F) - (°F - °F)}{\log \frac{(°F - °F)}{(°F - °F)}}$$

$$\Delta t_m = \frac{(600 - 145) - (250 - 40)}{\ln \frac{(600 - 145)}{(250 - 40)}}$$

$$\Delta t_m = 317 \text{ °F.}$$

Using the Dittus-Boelter equation (109a) the film coefficients were calculated for the cooling gas and the gas being cooled:

$$h_1 = 0.0225 \frac{k}{D} \left(\frac{Du\rho}{\mu} \right)^{0.8} \left(\frac{uC}{k} \right)^{0.3} \quad (8)$$

where

h_1 = film coefficient for the gas being cooled,
Btu/hr-sq ft-°F

k = thermal conductivity of condensed fluid,
Btu/hr-sq ft-°F/ft

D = diameter of the tube, ft

u = linear velocity of gas being cooled, ft/min

C = specific heat at constant pressure of the gas
being cooled, Btu/lb-°F

μ = absolute viscosity at bulk temperature, lb/hr-ft

ρ = density of material being cooled, lb/cu ft

then

$$h_1 = \left(\frac{\text{Btu-ft}}{\text{hr-sq ft-}^\circ\text{F}} \times \frac{1}{\text{ft}} \right) \left(\frac{\text{ft}}{1} \times \frac{\text{ft}}{\text{min}} \times \frac{\text{lb}}{\text{cu ft}} \times \frac{\text{hr-ft}}{\text{lb}} \right) \left(\frac{\text{Btu}}{\text{lb-}^\circ\text{F}} \times \frac{\text{lb}}{\text{hr-ft}} \times \frac{\text{hr-sq ft-}^\circ\text{F}}{\text{Btu-ft}} \right) = \text{Btu/hr-sq ft-}^\circ\text{F}$$

$$h_1 = 0.0225 \left(\frac{0.013}{0.054} \right) \left(\frac{0.054 \times \frac{15 \times 4}{(0.054)^2}}{0.0242} \right)^{0.8} \left(\frac{0.28 \times 0.0242}{0.13} \right)^{0.3}$$

$$h_1 = 9.5 \text{ Btu/hr-sq ft-}^\circ\text{F}.$$

The film coefficient for the cooling medium was then calculated using the Dittus-Boelter equation, however because the cooling medium was being heated, the exponent of the Prandtl number was changed to 0.4 from 0.3.

Substituting in equation 8:

$$h_2 = 0.0225 \times \left(\frac{0.13}{0.117} \right) \left(\frac{0.117 \times \frac{15 \times 4}{(0.054)^2}}{0.073} \right)^{0.8} \left(\frac{0.24 \times 0.073}{0.13} \right)^{0.4}$$

$$h_2 = 4.6 \text{ Btu/hr-sq ft-}^\circ\text{F.}$$

The overall coefficient of heat transfer was calculated neglecting the resistance of the tube wall.

$$U = \frac{1}{\frac{1}{h_1} + \frac{1}{h_2}} \quad (9)$$

where

U = overall coefficient of heat transfer,
Btu/hr-sq ft- $^\circ$ F

h_1 = coefficient of heat transfer for the material
being cooled, Btu/hr-sq ft- $^\circ$ F

h_2 = coefficient of heat transfer for the cooling
material, Btu/yr-sq ft $^\circ$ F

then

$$U = \frac{\text{Btu}}{\text{hr-sq ft-}^\circ\text{F}} + \frac{\text{Btu}}{\text{hr-sq ft-}^\circ\text{F}} = \frac{\text{Btu}}{\text{hr-sq ft-}^\circ\text{F}}$$

$$U = \frac{1}{\frac{1}{9.5} + \frac{1}{4.6}}$$

$$U = 3.1 \text{ Btu/hr-sq ft}^\circ\text{F.}$$

From the above calculated information the condenser cooling area required was calculated as follows:

$$A = \frac{q}{U \Delta t_m} \quad (10)$$

where

A = the required cooling surface area, sq ft

q = the heat to be removed from the hot material to the cooling material, Btu/hr

U = the overall heat transfer coefficient, Btu/hr-sq ft-°F

t_m = logarithmic mean temperature difference, °F

then

$$A = \frac{\text{Btu}}{\text{hr}} \times \frac{\text{hr-sq ft-}^\circ\text{F}}{\text{Btu}} \times \frac{1}{^\circ\text{F}} = \text{sq ft}$$

$$A = \frac{1575}{3.1 \times 317}$$

$$A = 1.6 \text{ sq ft.}$$

For construction purposes the length of pipe required for the inner tube was determined as follows:

$$L = \frac{A}{S_1} \quad (11)$$

where

L = length of pipe for required surface area, ft

A = required cooling surface area, sq ft

S₁ = internal surface area of 3/4-inch pipe, sq ft/ft

then

$$L = \frac{\text{sq ft}}{1} \times \frac{\text{ft}}{\text{sq ft}} = \text{ft}$$

$$L = \frac{1.6}{0.196}$$

$$L = 8.2 \text{ ft.}$$

Calculation of Per Cent Yield of Shale Oil. The yield of shale oil was determined from the difference in the loss of the raw shale charge and the loss of the retorted shale in a modified Fischer retort. The data obtained from retorting minus 16 plus 28 mesh shale are used for the following example:

$$P = \frac{R}{O} \times 100 \quad (12)$$

where

P = yield of shale oil on retorting in fluidized retort, per cent

R = shale oil remaining in retorting, grams/100 grams

O = shale oil in raw shale, grams/100 grams

then

$$P = \frac{R}{100 \text{ gm}} \times \frac{100 \text{ gm}}{O} \times 100 = \text{per cent}$$

$$P = \frac{10.59}{17.31} \times 100$$

$$P = 50.5 \text{ per cent.}$$

Calculation of Specific Gravity and A.P.I. Gravity of Shale Oil Samples. The specific gravity bottle was first calibrated with freshly distilled water and the volume of the bottle determined. An oil sample was then placed in the bottle and the weight of the bottle and oil determined. From these weights the specific gravity of the shale oil was determined. The data obtained from the shale oil on retorting minus 16 plus 28 mesh oil shale are used in the following example:

$$S = \frac{W - B}{H - B} \quad (13)$$

where

S = specific gravity of shale oil sample, no units

W = weight of pycnometer and shale oil sample, gm

H = weight of pycnometer and distilled water, gm

B = weight of pycnometer, gm.

Thus, from test one,

$$S = \frac{W - B}{H - B}$$

$$S = \frac{21.0360 - 11.3994}{21.3911 - 11.3994}$$

$$S = 0.964.$$

For use in determining the characterization factor of the shale oil samples the specific gravity of the samples were converted to A.P.I. by the following relationship:

$$\text{Degrees A.P.I.} = \frac{141.5}{\text{Sp. Gr.}} - 131.5$$

then

$$\text{Degrees A.P.I.} = \frac{141.5}{0.964} - 131.5$$

$$\text{Degrees A.P.I.} = 15.2.$$

Determination of the Kinematic Viscosity. Using the constant determined for the viscosity tube used and the time of efflux for the shale oil sample the kinematic viscosity of the shale oil was determined as follows, using the efflux time of the shale oil obtained on retorting minus 16 plus 28 mesh oil shale:

$$V = ct \quad (14)$$

where

V = kinematic viscosity, centistokes

c = calibration constant, centistokes/second

t = time of efflux, seconds

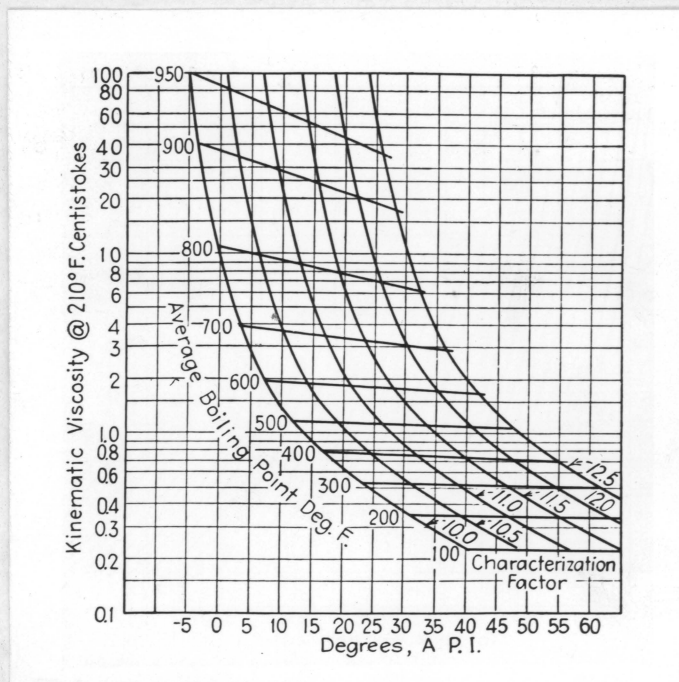
then

V = centistokes/second x seconds = centistokes

V = 0.2033 x 60.9

V = 12.18 centistokes.

Determination of Characterization Factor. The characterization factor was determined from Figure 17, page 120, by use of the A.P.I. gravity, in degrees, and the kinematic viscosity, in centistokes. The intersection of the points on the graph was taken as the characterization factor. For the test using oil shale of minus 16 plus 28 mesh the degrees A.P.I. was found to be 15.2, and the kinematic viscosity was determined to be 12.18 centistokes. Thus by the use of Figure 17, the characterization factor was found to be 11.2.



**Figure 17. Relation of Kinematic Viscosity and
A.P.I. Gravity to the Characterization
Factor and Average Boiling
Point of Crude Petroleum**

Nelson, W.L.: "Petroleum Refinery Engineering," p. 147.
McGraw-Hill Book Company, New York, N. Y., 1949.
3rd ed.

IV. DISCUSSION

A discussion of the design and construction of the retorting unit for retorting oil shale in a fluidized bed is included in the following paragraphs. Factors affecting the method of operation on the retorting unit as well as the method of operation of the fluidisation system as designed and constructed are discussed. The experimental results obtained are discussed with reference to the effect of the oil shale particle size on the A.P.I. gravity, kinematic viscosity, and average boiling points of the shale oil from the retort. The effect of the size of the oil shale retorted on the uncondensable gas yield as well as the yield of shale oil is noted. Recommendations regarding modifications of retorting equipment and operating procedure are advanced for use in further study. The experimental limitations imposed on this investigation are also listed.

Discussion of Results

Observations made during the investigation of the fluidized retorting of oil shale are discussed in the following paragraphs as to the design and construction of the fluidisation unit, the operating procedure, and the results obtained.

Discussion of the Design and Construction of the Retorting Unit. The unit for retorting oil shale was constructed in

sections for ease of assembly. These separate sections are discussed in the following paragraphs; included are the fluidized retort, heating section, disengaging section, cyclone separator, condensers, gas-circulating section, gas-measuring section, brine-circulation system, and control units.

Fluidized Retort. The fluidization section was originally constructed of four-inch black iron pipe with screwed flanges. It was found that the iron pipe shrunk away from the flanges after heating to retorting temperature and cooling. The only method of prohibiting the leakage of gas past the threads of the pipe and flange was to re-tighten the flange on the pipe. Use of slip-on-weld flanges at the top and bottom of the retort proved satisfactory.

The thermocouple and manometer taps on the retort, using brass ferrule type fittings screwed into black iron couples welded to the retort wall, were found to leak after heating to retorting temperature due to compression of the brass threads. Replacement of the brass fittings with fittings from stainless steel would improve this situation.

In the original construction a gas distribution plate was used which was constructed from a steel plate drilled $1/8$ inch on $3/8$ -inch centers. This distribution

plate gave very poor fluidization with a large amount of channeling. As shown in Figure 6, page 71, the final distribution plate consisted of a bonded porous silica plate mounted in a lathed flange, and supported with a steel plate drilled $1/4$ inch on $3/4$ -inch centers. This distribution plate gave very good fluidization, without evidence of channeling except at gas velocities approaching 15 cubic feet per minute at which slug flow took place.

In the original assembly of the retorting unit, bonded asbestos gaskets were used and on heating to the retorting temperature the retort was found to leak around the gasketed flanges. The unit was disassembled and it was found that the bonding material decomposed at the retorting temperature of 715°F . In the final construction corrugated metal asbestos gaskets were used and found to be satisfactory, however much care was necessary in tightening the flange bolts since uneven compression of the gasket caused the retorting gas to leak past the gasket. The use of a sealing compound was tried in an effort to prevent the gas leakage due to uneven compression or pitted flange surfaces, but it was found to give off solvent when heated to retorting temperatures, allowing the gas to pass the flange. The final method of preventing leaks at the flanges was to machine

the flange face and then prepare a gasket ridge on the flange face as shown in Figure 6. Additional precautions were also taken by tightening opposite bolts on the flange at the same time.

Gas-heating Section. In the original design and construction the gas-heating section was constructed from screwed flanges on two-foot sections of 8-inch diameter black iron pipe. The heating elements were wired through 1/2-inch black iron nipples, and welded into the side of the 8-inch pipe. The 1/2-inch nipples were packed with graphited asbestos valve packing, and a 1/2-inch bushing was screwed down over the heater leads, thus compressing the packing around the heating element leads. Thermocouples were inserted into the heating section over each heating element by use of a brass ferrule-type fitting which were screwed into holes drilled and tapped into the heating section wall. When heated to operating temperature, the screwed flanges, thermocouple fittings, and heating elements were found to leak at two pounds per square inch, gage pressure.

The heating section was finally constructed from a five-foot section of 8-inch black iron pipe with weld-neck flanges on each end. The heaters leads were inserted through 1/2-inch black iron nipples welded into the pipe wall at six-inch levels over the pipe length, as shown

in Figure 8, page 76. The heater leads were inserted through the nipples from the inside of the pipe and welded to the nipple. Monel wire column packing was placed between each heating element to give a larger heat transfer area. As shown in Figure 14 of the Appendix, a 2-1/2-inch gas transfer line to the fluidized retort from the heating section was attached to the bottom of the gas-heating section.

Originally the gas transfer line was constructed from 2-inch pipe joined with screwed fittings. The 2-inch gas line was attached to the bottom of the heating section by means of a drilled and tapped 2-inch pipe size hole in the center of an 8-inch cast iron blind flange. After heating, the gas transfer line was found to leak at 2 pounds per square inch, gage pressure. The gas transfer line was finally attached to the bottom of the gas-heating section by means of an 8-inch slip-on-weld flange which had a 3/4-inch thick mild steel plate welded in the open end. The 2-1/2-inch gas transfer line was then welded to the steel plate in a hole cut into the center of the plate. All fittings required to connect the gas transfer line to the retort were welded together.

In order to heat the gas transfer line, as well as to supply extra heat to the retorting gas, the gas transfer

line had strip heaters attached to the exterior walls of the pipe. The gas transfer line was not heated in the original design and the heat required to heat this line to retorting temperature materially increased the time required to reach retorting temperature in the retort.

Even with the additional heaters, it was found that at maximum air-flow rate the maximum temperature of the fluidizing gas was limited to 800 °F. The gas-heating section should be redesigned and the fluidizing gas heated by gas flame against return bend coils. Redesign of the gas-heating section would reduce the heating time for the system and increase the value of the retorting system for further use.

The heating section was insulated with a 2-inch layer of 85 per cent insulation, which proved inadequate and allowed a large amount of heat to escape from the wall of the heating section to the room. Additional insulation should be used on the gas-heating section.

Disengaging Section. The original disengaging section was designed two feet long and 8 inches in diameter. It was constructed from 8-inch diameter black iron pipe with screwed flanges on each end. The disengaging section was connected to the top of the retorting section by means of a 4 x 8-inch diameter

cast iron flanged reducer. The top of the disengaging section was joined to the cyclone separator through a 3/4-inch black iron line. The 3/4-inch black iron pipe was attached to the top of the disengaging section by means of a 3/4-inch hole drilled and tapped into the center of a blind flange belted to the top of the disengaging section. When heated and cooled, the disengaging section leaked through the threaded connections at 2 pounds per square inch, gage pressure.

Finally the disengaging section was constructed from a two-foot section of 8-inch diameter black iron pipe with slip-on-weld flanges on each end of the pipe. The disengaging section was connected to the top of the retorting section by means of a 4 x 8-inch diameter cast iron flanged reducer. The cyclone separator was connected to the disengaging section by means of a 3/4-inch black iron pipe attached to the top of the disengaging section. A 3/4-inch hole drilled and tapped into the center of an 8-inch blind cast iron flange provided an outlet for the gases from the disengaging section to the cyclone separator. The blind flange was gasketed and belted to the top flange of the disengaging section. Similar gasketing experiences were had with the disengaging section, as with the gas-heating and retorting section.

The disengaging section was found, from previous experience, to require heating in order to prevent

condensation of the retorted shale oil on the sides of the section. At first heating was attempted by the use of resistance heating wire passed through ceramic holders. The heater thus constructed was held against the wall of the disengaging section by means of holders welded to the pipe wall. This method of heating proved unsatisfactory because the heaters did not possess the capacity for heating the disengaging section, possibly because of the method of insulating the heaters from the pipe wall. The heaters also had a tendency to burn out quickly. The disengaging section was finally heated by means of heaters in ceramic plates attached to the disengaging section wall by studs through the plates and welded to the wall. These heaters were very satisfactory and no condensation was observed on the inside of the disengaging section walls.

Cyclone Separator. The original cyclone separator was constructed from 20-gage sheet metal and was found to be unable to stand a pressure of ten pounds per square inch, gage. The cyclone finally used was constructed from 1/8-inch mild steel plate, as shown in Figure 9, page 60. The cyclone separator proved satisfactory in separating solids from the gases and was found to separate all solids at gas velocities below 15 cubic feet per minute, but above this velocity particle sizes below 100 mesh (Tyler screen scale) were transported into the first condenser.

The original cyclone separator was heated by means of heating elements in ceramic cylinders attached to the cyclone wall. As with the disengaging section the heaters did not prove satisfactory. The final cyclone was heated by heating elements in ceramic plates attached to the separator wall by means of studs through the plates attached to the cyclone walls. These elements proved to be very satisfactory, and no condensation of oil was noted in the solids removed from the separator. The present port for removing solids was very unsatisfactory, because the 3/4-inch outlet provided for this purpose was too small and became clogged easily. At least a four-inch outlet should be provided so that after re-torting the unit could be cleaned and the cyclone walls observed for any condensation.

Shale Oil Condensers. The condensers were constructed as simple double-pipe heat exchangers operating countercurrently. The first condenser was designed to be cooled by the gases returning from the brine cooled condenser, and when calculated for the area required it was found that at least 8 feet of 3/4-inch copper tube would be needed to give the required area. The condenser was then redesigned as two condensers each providing five feet of cooling surface, because of head room limitations. The water-cooled and brine-cooled

condensers were calculated to require approximately five feet of 3/4-inch pipe to provide the cooling surface necessary to cool the gas to the desired temperature. The gas-cooled condensers were designed to operate at a minimum temperature of 140 °F. The water-cooled condenser was designed to operate at a minimum temperature of 60 °F, and the brine-cooled condenser was designed to operate at a minimum temperature of 40 °F. In four tests it was found that at a retort temperature of 715 °F, the first gas-cooled condenser had an incoming gas temperature of 708 °F and a leaving gas temperature of 496 °F. The second gas-cooled condenser had an incoming gas temperature of 460 °F and a leaving gas temperature of 314 °F. If compared to the desired minimum operating temperature of 140 °F, it can be seen that the calculation of condenser size did not hold in this case. The gas-cooled condensers definitely require a larger surface area to accomplish the cooling desired.

The water-cooled condenser had approximate entering and exit gas temperatures of 296 °F and 134 °F respectively. This is substantially above the desired minimum operating temperature of 60 °F. The temperature of the entering gas to the condenser however is 156 °F above the design gas inlet temperature of condenser. The error

in design is therefore believed to be in the gas-cooled condensers, although constructed two feet longer than calculated necessary.

The condensers were all constructed in a similar manner and the detail of construction is shown in Figure 10, page 82.

Considerable difficulty was encountered in attaching the shale oil receivers to the condensers. The use of a yoke to hold the receiver in contact with the receiver header was satisfactory but the flange holding the yoke was constructed from copper plate and was deformed easily, when the yoke was tightened against the receiver. Construction of the flange from steel would eliminate this problem.

A change of construction material for the condensers was indicated because the hydrogen sulfide in the retort gases reacted with the copper pipe used for construction of the condensers. Fouling of the tubes, particularly in the water-cooled and brine-cooled condensers was observed, however all traces of the material was removed by washing with water and brushing with a long handled brush.

Gas Circulation System. Gas circulation through the fluidised system was accomplished by means of a sliding-vane pump. The gas pump was internally

lubricated and an oil separator was provided on the outlet side of the pump, however it was observed that lubricating oil was still passing into the gas line to the heating section. In an effort to correct this a cooling coil of 1/4-inch copper tube was wound around the oil separator, and water circulated through the coil. It was observed that when the retort was at 715 °F, the gas returning to the gas pump was approximately 200 °F. The gas pump efficiency was lowered by circulating the hot gas. The possibility of lubricating oil being evaporated and carried into the gas-heating section also existed. The gas pump should be placed in the exit gas line of the brine cooled condenser. This would eliminate the reduction in efficiency of the pump caused by circulating hot gas as well as reduce the possibility of evaporation of the lubricating oils from the pump.

The maximum capacity of the gas pump supplying gas at atmospheric pressure was 18 cubic feet per minute. Preliminary observations of the fluidization characteristics of shale in a 4-inch diameter column indicated that a gas volume greater than 18 cubic feet per minute would be required for fluidization of shale larger than 16 mesh (Tyler screen scale). Study of the retorting of oil shale of larger than 16 mesh was prohibited by

the capacity of the gas pump. Replacement of the present pump would be desirable both from the standpoint of capacity, and method of lubrication.

Gas Measuring Section. The original orifice designed for measuring the gas flow through the retorting unit had an orifice diameter of $5/16$ inch. The pressure drop across the orifice was approximately 3 pounds per square inch, gage, which was higher than the pressure drop across the entire system. The orifice was reconstructed with an orifice diameter of $1/2$ inch. The pressure drop across the orifice as finally designed was less than one pound per square inch, gage.

Leads from the orifice to the manometers were first constructed from $1/4$ -inch black iron pipe, however the orifice lines leaked, and were replaced with $1/8$ -inch copper tubing. The copper tubing leads were easily clogged and resulted in erroneous readings on the manometer, during retorting. The clogging of the lines could possibly be eliminated by use of larger diameter tubing.

The orifice was calibrated in position by use of a gas meter with a one-inch inlet and outlet. The orifice was constructed of $3/4$ -inch black iron pipe and screwed $3/4$ -inch flanges.

Discussion of the Conditions of Retorting. The conditions of retorting imposed on this investigation were retorting temperature of 715 °F, fluidizing gas of carbon dioxide, retorting pressure of 10 pounds per square inch, retort gas containing less than one per cent oxygen, oil shale charge weight of 16-1/4 pounds, and retorting time of one hour.

The retorting temperature of 715 °F was chosen as the retorting temperature because it was 100 °F higher than the temperature at which the organic material of oil shale begins to decompose and distill from the oil shale. According to Gardner⁽²⁸⁾ shale oil begins to distill from oil shale between 600 and 650 °F, and all kerogen is converted to oil if retorting is continued to approximately 900 °F.

Carbon dioxide was selected as the fluidizing medium because it will not support combustion and has less tendency to combine with the materials used for construction of retorting unit. Nitrogen was originally selected for the fluidizing gas, however the gas heating section was observed to have a black coating of material on the inside indicating that possibly nitrides were forming.

The operating pressure of ten pounds was selected because the volume of the gases at atmospheric pressure in the system was approximately twice that of the system, and the density of the fluidising gas was increased sufficiently to increase the heat capacity of the gas to give satisfactory operation of the retort.

The charge weight was determined by filling the retort to a bed level of 28 inches, the maximum deemed feasible for operation, as this allowed 8 inches for expansion of the bed level. The weight of oil shale of plus 16 and minus 28 mesh required to fill the retort to the bed level of 28 inches was used as the charge weight in all tests. The charge weights of tests two and four varied from 16-1/4 pounds minus 2 ounces and minus one ounce, respectively.

Discussion of Shale Oil Analysis. The shale oil recovered from retorting was extracted in soxhlet extractor to remove any solids from the oil which would interfere with the specific gravity or kinematic viscosity determinations. The extraction was performed with ethyl ether.

The shale oil was analysed for specific gravity at 60 °F according to the method described in the American Society of Testing Materials designation D 70-27^(106a). The specific gravity was then converted to degrees A.P.I. for determining the characterization factor of the shale oil.

The kinematic viscosity of the shale oil was determined at 210 °F according to the method described in the American Society of Testing Materials designation D 445-46 T. The viscosity was difficult to determine because the color of the shale oil adhering to the sides of the pipet obscured the meniscus of the shale oil flowing down the pipet. Four

determinations were made and an average of the four taken as the kinematic viscosity.

Analysis of Oil Shale. The amount of shale oil available in the oil shale charges of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh varied from 17.3 to 17.1, 15.4, and 15.0 weight per cent respectively. Duplicate tests for available oil in samples of each particle size range were made using the Fischer Retort Method⁽³¹⁾ and the results obtained agreed to within \pm 0.2 per cent. These differences in shale oil content of the samples of various particle sizes may or may not be significant. It would seem, however, the differences are somewhat significant when the method of preparing the samples of the various particle sizes is considered. The shale as received was passed through a jaw crusher and screened. Sample of minus 16 plus 28, minus 28 plus 70, and minus 70 plus 100 mesh were obtained from this operation. The material of minus 100 plus 200 mesh was obtained by grinding, in a rod mill, the oversize material remaining on the 16-mesh screen. Since oil shale is a non-homogeneous, striated material it would be expected to exhibit the property of being more easily reduced in size with increasing retortable material and possibly explains the relative difference in oil content between the largest and smallest particle size ranges. Again, the differences may not be so significant when the procedure

used in the method of analysis is considered. The oil content was determined on the various particle size ranges of samples as such without attempting to reduce all samples to same particle size range. Just what effect the particle size range has in such determinations is not known.

Method of Gas Analysis. The gas was analyzed according to standard procedure for gas analysis. The hydrogen and ethane was determined by combustion with oxygen, and calculation of the combustible material into hydrogen and ethane.

The gases, before being drawn into the orsat apparatus, were dried by passing through an absorption bulb packed with a layer of calcium chloride on the bottom, then a layer of calcium chloride on top of the cupric sulphate. The hydrogen sulphide in the gas reacted with the cupric sulphate to give copper sulphide and water. The water from the reaction was absorbed on the calcium chloride above the cupric sulphate. The hydrogen sulphide in the gas was determined from the increase in weight of the absorption bulb and the volume of the gas passed through the bulb, which was measured by use of the orsat apparatus. The gas furnished to the orsat apparatus was free from water vapor and hydrogen sulphide.

The analysis of the gases can only be accurate within 0.2 per cent as the gas-measuring bulb was calibrated in 0.2 milliliter increments. All analysis were corrected for atmospheric pressure and temperature.

Discussion of the Results of the Fluidized Retorting of Oil Shale. The results of the fluidized retorting of oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale) in a 4-inch diameter fluidized bed at 715 °F for one hour are discussed in the following paragraphs. The effect of oil shale particle size on the yield of shale oil and non-condensable gases, the A.P.I. gravity, the kinematic viscosity, the mid-boiling point, the average boiling point, and the characterization factor of the shale oil are discussed.

Effect of Oil Shale Particle Size on the Yield of Shale Oil. The yield of the shale oil decreased with the size of the particles of oil shale retorted. For particle sizes of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), the shale oil yields of the available shale oil were 50.5, 47.3, 40.9, and 39.4 per cent, respectively.

It is believed that the decrease in shale oil yield was due to the agglomeration of the oil shale in the retort and cracking of the shale oil into gases. With smaller particle sizes, better heat transfer would be expected resulting in cracking into gases. Agglomerated shale was found in the retorted shale of tests using shale of minus 70 plus 100 and minus 100 plus 200

mesh. In the test employing minus 70 plus 100 mesh, agglomerated oil shale lumps were found less than 1/2 inch in diameter. Lumps as large as one inch in diameter were found in the minus 100 plus 200 mesh retorted oil shale. The lumps found were easily broken, and no evidence of fixed carbon was observed inside the lumps as observed on the surface of the lump. No attempt was made to analyze the material inside the lump, however a representative sample was taken from the retorted shale and analyzed for shale oil content.

Reduction of the shale oil yield by conversion of the shale oil into non-condensable gases by cracking is apparent from the increase in the volume of gases generated of 0.67, 0.72, 1.01, and 1.2 cubic feet for oil shale particle sizes of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh. Cracking of the shale oil is also exhibited by the decrease in mid-boiling point, average boiling point, and kinematic viscosity of the shale oil with shale oil particle size. Some of the reduction in kinematic viscosity, mid-boiling point, and average boiling point of shale oil may be due however to the incomplete retorting of shale oil leaving the heavier, harder to retort material in the oil shale. From analysis of the retorted

shale, the shale oil remaining in the oil shale was 8.56, 8.98, 9.1, and 10.6 per cent for oil shale particle size of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh. From the data obtained, no definite conclusions can be reached as to the exact effect of shale oil particle size on the yield of oil from shale, however it can be stated that a reduction of oil shale particle size affects a reduction in the yield of shale oil, mid-boiling point, average boiling point, and viscosity of the shale oil obtained, as well as increase in the A.P.I. gravity and volume of non-condensable gases generated.

Additional studies should be made to ascertain the effect of oil shale particle size as well as the effect of temperature and retention time on the yield of shale oil.

Effect of Oil Shale Particle Size on the Yield of Non-condensable Gases from the Retort. The non-condensable gas generated in the retort increased with decreasing particle size of oil shale retorted. The non-condensable gas was 0.67, 0.72, 1.01, and 1.2 cubic feet for oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), respectively. The increase in volume

of non-condensable gases could be due to several reasons. Possibly the method of preparation of the oil shale for the tests as previously explained had some effect on the yield of gas from the retort.

The indication is that the increase in gas volume was due to cracking of the shale oil rather than the available shale oil or the kind of shale oil in the shale tested. The hydrogen content was 0.6, 0.6, 1.4, and 10.2 per cent of the gas volume, and the hydrocarbons, reported as ethane, was 9.0, 12.0, 14.6, and 22.4 per cent of the gas volume for oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh, respectively. The increase in gas volume can definitely be attributed to the increase of hydrogen and ethane in the system, indicating that cracking undoubtedly took place in the retort.

Effect of Oil Shale Particle Size on the Degrees A.P.I. of Shale Oil. As indicated from the increase in hydrocarbon fractions in the retort gases, the shale oil recovered from the retorting unit condensers increased in degrees A.P.I. with decreasing particle size. For test one, using oil shale of minus 16 plus 28 mesh, the shale oil had an A.P.I. gravity of 15.2 degrees. The A.P.I. gravity of the shale oil increased to 17.9, 18.6, and 22.4 degrees for tests two, three, and four

using oil shale of minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh oil shale as the material retorted. The decrease in shale oil yield retorted from the oil shale could be considered as one reason for the increase in A.P.I. gravity of the shale oil. The shale oil available in the oil shale after retorting was 8.56, 8.98, 9.1, and 10.6 per cent by weight for oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh, respectively. The heavy fractions of the shale oil most probably were retained in the shale causing a decrease in A.P.I. gravity of the recovered oil.

It is believed that cracking of the shale oil, rather than selective retorting due to method of preparation of the samples, took place in the fluidized bed. From the data on the shale oil retained on the oil shale, it is seen that the greatest amount, 10.6 per cent by weight, was retained in minus 100 plus 200 mesh oil shale. The greatest increment between the amounts of shale oil retained on the oil shale was between the oil shale of minus 70 plus 100 and minus 100 plus 200 mesh, respectively. The greatest change in A.P.I. gravity occurred between the minus 70 plus 100 and minus 100 plus 200 mesh oil shale, with the A.P.I. changed from 18.6 to 22.4 degrees, respectively. This would tend to indicate that the

material retained in the oil shale had a large effect on the A.P.I. gravity however, on observation of the values obtained for minus 16 plus 28, and minus 28 plus 70 mesh oil shale, the per cent shale oil in the oil shale after retorting increased 0.42 per cent between tests, with an increase of 2.7 degrees in the A.P.I. gravity of the shale oil. The increase in the A.P.I. gravity could not be due entirely to the oil not retorted from the shale.

Effect of Oil Shale Particle Size on the Kinematic Viscosity of Shale Oil. The kinematic viscosity of the shale oil obtained was found to decrease with oil shale particle size. Kinematic viscosities of 12.18, 4.64, 2.94, and 1.66 centipoises were obtained for oil shale particle sizes of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh, respectively. The decrease of kinematic viscosity possibly could be attributed to a number of factors other than the decrease in particle size of the oil shale. As previously discussed under the effect of particle size on the yield of shale oil, the type of available retortable material in the oil shale could have been a contributing factor to the decrease of the kinematic viscosity, however on comparison of the available oil, 17.3, 17.1, 15.4, and 15 weight per cent for oil shale

particle sizes of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh, with the kinematic viscosity obtained for the shale oil, no correlation is present.

The largest decrease in kinematic viscosity occurs between tests employing minus 16 plus 28 and minus 28 plus 70 mesh oil shale particles as the material being retorted. The decrease in kinematic viscosity was 7.54 centipoises while the decrease in available oil from the shale was only 0.2 weight per cent. The increase in quantity and composition of gases generated in the retort bears no relationship to the decrease in viscosity. It is believed that cracking of the shale oil occurred to such an extent in the test employing minus 28 plus 70 mesh oil shale, that very little gas was produced but a definite reduction of kinematic viscosity took place.

Effect of Oil Shale Particle Size on the Mid-boiling Point of the Shale Oil. A decrease of oil shale particle size was found to affect a reduction of the mid-boiling point of the shale oil. For retorting oil shale particle sizes of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh the mid-boiling points of the shale oil were 510, 500, 482, and 380 °F, respectively. As discussed in the

foregoing paragraphs, the shale oil content of the oil shale retorted might have caused the reduction of the shale mid-boiling point, however if this were the only factor, it is believed that the reduction of the mid-boiling point would not be nearly so great. Tests using minus 70 plus 100 and minus 100 plus 200 mesh oil shale had approximately 100 °F difference in the mid-boiling point of the shale oil recovered, while the per cent of shale oil available from the oil shale was approximately the same 15.4 and 15.0 per cent, respectively. The test employing oil shale of minus 28 plus 70 had an available shale oil yield of 17.1 per cent, 2.7 per cent greater than oil shale of minus 70 plus 100 mesh, yet the mid-boiling points only differed 18 °F.

Cracking of the shale oil in the retort is indicated as the possible factor, causing reduction of the mid-boiling point of the shale oil obtained. Although no appreciable increase in the volume of the gases from the retort indicates cracking of the shale occurring in the test using minus 100 plus 200 mesh oil shale, the hydrogen increases sharply to 10.2 per cent from 1.4 per cent for the previous test using minus 70 plus 100 mesh oil shale. The hydrocarbon portion, reported as ethane, of the retort gases increased from 14.6 per cent for oil shale of minus 70 plus 100, to 22.4 per cent for oil shale of minus 100 plus 200 mesh. This

indicates that cracking definitely took place although it is not known to what degree the shale oil was converted to non-condensable hydrocarbons.

Effect of Oil Shale Particle Size on Characterization Factor. No correlation could be found between the characterization factor of the shale oil retorted from the oil shale in the fluidized bed. Characterization factors of 11.2, 11.4, 11.2, and 11.0 were obtained for oil shale particle sizes of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale). It is significant however that for shale oil, the characterization factor was approximately the same, which would be expected for crude petroleum from the same source.

Recommendations

The following are recommendations evolving from observations made during the construction and operation of a fluidized system for the retorting of oil shale. These recommendations include equipment modifications and changes in operating conditions.

Heating Section. In the present design, the temperature of the fluidizing gas is limited to 1000 °F. It is recommended that the heating section be redesigned using a gas flame against pipe coils. With the proper design, the temperature of the fluidizing medium would be limited to approximately 2400 °F, and the delay of electrical heaters would be eliminated. The temperature of the fluidizing medium could be controlled by controlling the number of gas heaters in operation as well as the flow of the combustion gas.

Retorting Section. The present retort is limited to a charge weight of approximately twenty pounds of oil shale, and has the disadvantage of having to be removed from the system to be loaded and unloaded. The present batch system of retorting definitely limits the charge in the retort, the yield of shale oil, and the length of retorting time, and thus it is very difficult for the system to reach equilibrium. It is suggested that a continuous system of retorting be designed using the fluidized solids technique. The recommended

design, shown in Figure 18, page 149, includes a shale feeding system, oil shale drawoff, fluidized retort, and fixed carbon burn-off chamber.

Cyclone Separator and Disengaging Section Heating

System. The present heaters on the cyclone separator and disengaging section, used to prevent condensation of the shale oil vapors on the sides of equipment, proved satisfactory. The method of controlling the heaters with rheostats was found too difficult and time consuming. It is recommended that the cyclone separator and disengaging section heaters be controlled separately by use of automatic controllers.

Condensers. During operation of the fluidized retorting system, it was observed that a small amount of low-boiling shale oil was carried into the return line to the surge tank. It is suggested that the last two condensers be made brine-cooled condensers. Therefore the second condenser should be water-cooled, and the first condenser cooled by the return gas from the brine-cooled condensers.

In the present receivers, the flow of the carrier gas over the condensed product caused evaporation of the material. As shown in Figure 18, page 149, it is suggested that the receivers be constructed from stainless steel with a cooling jacket which could be connected to the brine circulation system. Cooling of the receiver contents by this method would prevent evaporation of condensed material.

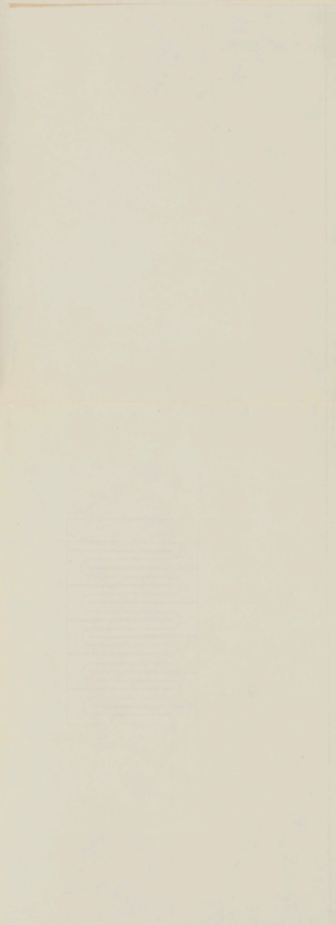
Figure 18

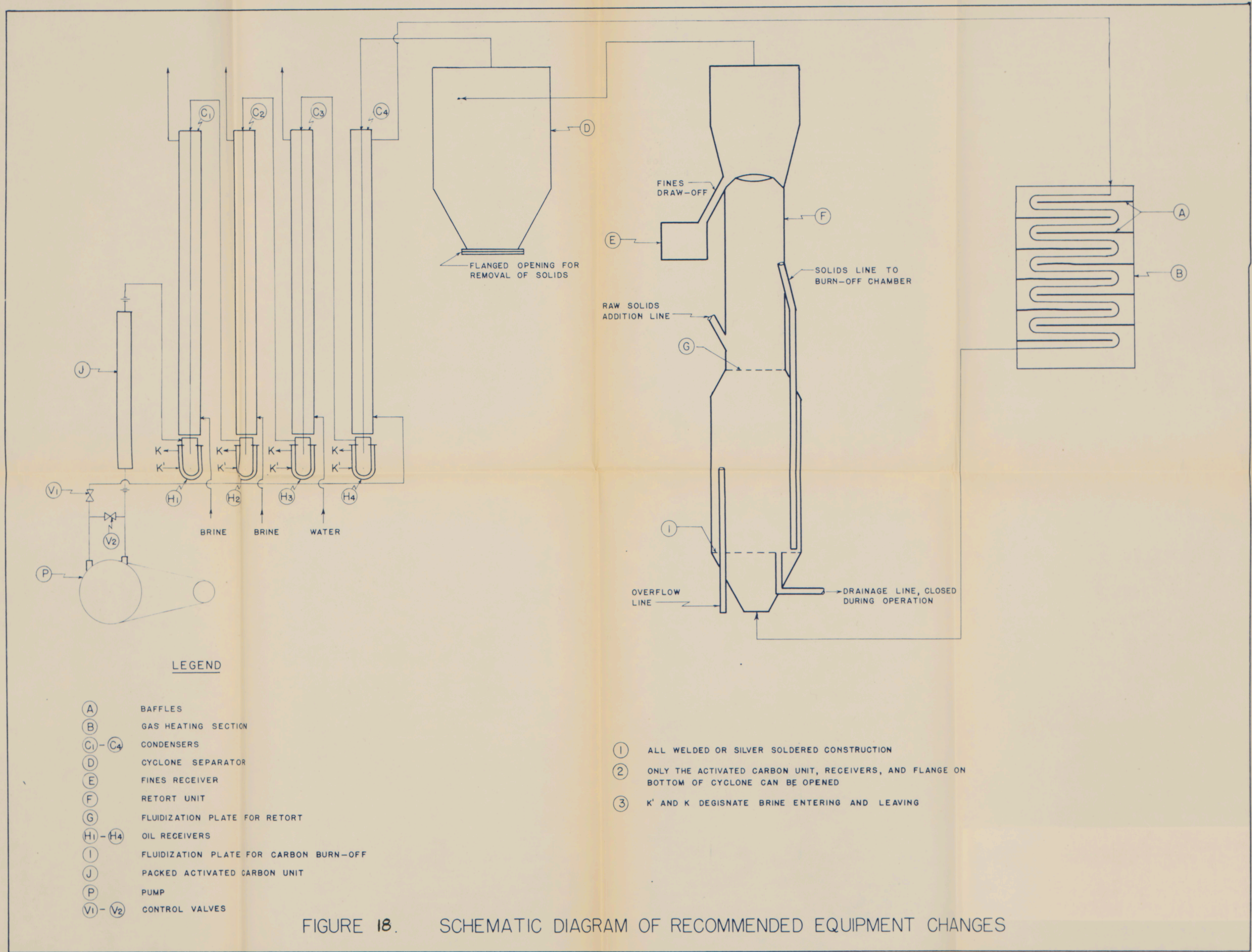
Schematic of Recommended Changes in Retorting Unit

(see pocket on back cover)

Figure 18

Schematic of Recommended Changes in Retorting Unit





LEGEND

- (A) BAFFLES
- (B) GAS HEATING SECTION
- (C₁ - C₄) CONDENSERS
- (D) CYCLONE SEPARATOR
- (E) FINES RECEIVER
- (F) RETORT UNIT
- (G) FLUIDIZATION PLATE FOR RETORT
- (H₁ - H₄) OIL RECEIVERS
- (I) FLUIDIZATION PLATE FOR CARBON BURN-OFF
- (J) PACKED ACTIVATED CARBON UNIT
- (P) PUMP
- (V₁ - V₂) CONTROL VALVES

- (1) ALL WELDED OR SILVER SOLDERED CONSTRUCTION
- (2) ONLY THE ACTIVATED CARBON UNIT, RECEIVERS, AND FLANGE ON BOTTOM OF CYCLONE CAN BE OPENED
- (3) K' AND K DESIGNATE BRINE ENTERING AND LEAVING

FIGURE 18. SCHEMATIC DIAGRAM OF RECOMMENDED EQUIPMENT CHANGES

Receivers for Condensed Products. The present copper flange on the receiver headers, used for receiver yoke attachment and gasketing, did not possess enough strength to enable the proper amount of pressure to be exerted by the yoke on the receiver for sealing without deforming the flange. It is suggested that mild steel be used for this flange.

Activated Carbon Trap. In order to prevent the possibility of entrainment of shale oil from the brine cooled condensers into the return gas line, and to reduce the possibility of shale oil not being condensed or removed from the gas returning to the heating section, it is recommended that an activated carbon trap be placed in the return gas line immediately after the brine cooled condenser, as shown in Figure 18, page 149

Brine Cooling System. The present system of cooling, brine solution with ice, is unsatisfactory and costly. In the event of tests of longer duration than eight hours, the ice requirement of 200 pounds would be increased. It is suggested that a refrigeration system be installed for cooling the brine solution.

Gas-pumping Section. The present gas pump is internally lubricated, and the possibility of oil vapor or entrained lubricating oil being accounted for as shale oil exists. As a preventive measure, an oil filter with cooling coils

was installed on the exit line of the gas pump, however no method of measuring the filters effectiveness was employed. It is recommended that a water-cooled pump be installed.

The pump was installed in the gas return line from the surge tank to the heating section. The temperature of the gas to the pump was approximately 265 °F at the height of retorting. The pump was circulating a hot gas thus enhancing the possibility of lubricating oil vaporization as well as reducing the efficiency of the gas pump. It is recommended that the gas pump be installed, as shown in Figure 18, page 149 in the gas line from the suggested activated carbon unit.

Auxiliary Equipment. The present manometer leads of 1/8-inch copper tube proved to be unsatisfactory in that the lines plugged and prevented transfer of the pressure to the manometers. It is suggested that 1/4-inch copper tube be used as manometer lines to the orifice and to the fluidised bed, with a trap to remove any material which might plug the manometer lines.

Thermocouple and Manometer Connections. After heating and cooling of the fluidisation unit it was observed that the brass copper-tube to pipe adapters, used to connect the thermocouples to the equipment as well as those used as pressure connections, were deformed by heat. It is recommended that stainless

steel copper-tube to pipe adapters be used to make all connections of the above type.

Lagging. During operation of the fluidized retort a large amount of heat was lost to the atmosphere. The present insulation of one layer of 85 per cent magnesia was unsatisfactory in conserving heat and it is suggested that either high temperature insulation or blown glass be employed as insulation for the retort.

Construction of Retorting Unit. The construction and operation of the fluidization unit was hampered by the low ceiling of the area in which the unit had to be constructed. It is recommended that the unit be reconstructed in an area with no less than a 25-foot ceiling. A catwalk constructed 8 feet from the floor would increase the efficiency and safety of construction, operation, and maintenance of the fluidization unit.

Considerable difficulty was encountered in construction of a leak-proof retorting system. At the temperature of operation, 700 ° to 1200 °F, it was found that screwed and flanged joints leaked gas on cooling to ambient temperature and being pressurized above atmospheric pressure. Therefore it is recommended that the retorting system be reconstructed as set forth in the foregoing paragraphs, using all welded joints.

Variation of Fluidized Bed Temperature. It would be desirable to study the effect of retorting temperature on the quality and quantity of shale oil from oil shale of various particle sizes. Operation of the fluidized retort at temperatures from 800 ° to 1000 °F would provide additional data on the effect of oil shale particle size on the characterization factor of the shale oil obtained.

Extension of Retorting Time. The maximum amount of shale oil recovered from the available shale oil was 50.5 per cent. It is recommended that the effect of retorting time be studied to determine its effect on shale recovery.

Modified Fischer Retort. The results obtained from the analysis of the oil shale in the modified Fischer retort varied from the expected and is recommended that the effect of oil shale particle size on the Fischer retort analysis be studied.

Method of Preparation of Oil Shale. The method of preparation of the oil shale seemed to have some bearing on the amount of available shale oil in the oil shale. It is recommended that samples of oil shale for retorting be prepared by carefully crushing or grinding the oil shale to the desired particle size range, using a portion of the oil shale as received for each sample rather than attempting to prepare all retorting samples from one oil shale sample of the material received.

Limitations

The limitations imposed on the investigation of the fluidized retorting of oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale) are listed below.

Fluidization Chamber. The fluidization chamber used was constructed of a three-foot length of a 4-inch nominal diameter black iron pipe.

Fluidized Solid. The fluidized solid consisted of oil shale ground to minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale). The oil shale was obtained from the Bureau of Mines, Rifle, Colorado in cooperative agreement with the United States Department of the Interior. The charge weight for each test was 16-1/4 pounds.

Fluidizing Medium. Carbon dioxide at 715 °F was utilized as the fluidizing medium to prevent combustion of shale oil during retorting.

Gas Velocities. The gas velocities used for fluidizing the oil shale were 14.6, 10.5, 7.0, and 5.0 cubic feet per minute for oil shale particle sizes of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale).

Gas Pressure. In all tests, the fluidization system was operated at 10 pounds per square inch, gage, gas pressure.

Time. The retorting unit was operated for one hour at a retort temperature of 715 °F under the above limiting conditions. The time for preheating the retorting unit to retorting temperature was limited to 3-1/2 ± 1/2 hours.

V. CONCLUSIONS

An evaluation of the results of the investigation of the fluidized retorting of oil shale for one hour in a 4-inch diameter fluidized bed using carbon dioxide at 715 °F as the fluidizing medium, oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale) as the solid in the fluidized bed, led to the following conclusions:

1. The shale oil yield was found to decrease with shale oil particle size. For retorting tests using oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), the shale oil yields were 50.5, 47.3, 40.9, and 39.4 per cent, respectively.
2. The A.P.I. gravity was found to increase with oil shale particle size. The A.P.I. gravity was 15.2, 17.9, 18.6, and 22.4 degrees for oil shale particle size of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale).
3. The kinematic viscosity was found to decrease with oil shale particle size. For oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100,

- and minus 100 plus 200 mesh (Tyler screen scale), the kinematic viscosity decreased 12.18, 4.64, 2.94, and 1.66 centipoises, respectively.
4. The mid-boiling point of the shale oil was found to decrease with a decrease in oil shale particle size. The mid-boiling points of the shale oil retorted from oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale) was 510, 500, 482, and 380 °F, respectively.
 5. The volume of gas generated in the retort increased with a decrease in the particle size of the oil shale retorted. The gas volume increased 0.67, 0.72, 1.01, and 1.2 cubic feet for oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), respectively.
 6. The per cent of hydrocarbons in the gas generated in the retort, reported as ethane, increased with a decrease in particle size of the oil shale retorted. The per cent of ethane in the retort gas was 9.0, 12.0, 14.6, and 22.4 per cent for oil shale particle size of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), respectively.

7. The per cent of hydrogen in the gas generated in the retort increased with a decrease in particle size. For oil shale particle size of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), the hydrogen was 0.6, 0.6, 1.4, and 10.2 per cent.
8. The oil shale particle size had no effect on the hydrogen sulfide in the retort gases. Hydrogen sulfide contents of 1.2, 1.3, 0.8, and 1.0 per cent were obtained for oil shale retorted of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), respectively.
9. Particle size of the oil shale retorted had no effect on the characterization factor of the shale oil obtained. Characterization factors of 11.2, 11.4, 11.2, and 11.0 were obtained for tests using oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), respectively.

VI. SUMMARY

Considerable work has been done on the economic production of shale oil from oil shale. Of the possible materials which could replace crude petroleum as used today, oil shale possesses the greatest possibilities since, when retorted, it produces a shale oil very similar to crude petroleum, and on refining the products produced are similar. Of the fuel resources available in the United States, 98.8 per cent is attributed to coal, 0.8 per cent to oil shale, 0.2 per cent to crude petroleum, and 0.2 per cent to natural gas.

The purpose of this investigation was to study the effect of oil shale particle size on the shale oil obtained from oil shale retorted in a fluidized bed. Oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale) was retorted at 715 °F in a 4-inch diameter retort 36 inches long using carbon dioxide as the fluidizing medium. A retorting unit consisting of a fluidizing retort, fluidizing gas-heating section, disengaging section, cyclone separator, condensers, a surge tank, gas circulation system, and temperature measurement and control equipment was designed and constructed.

The fluidizing gas-heating section was constructed from a flanged 5-foot section of 8-inch diameter black iron pipe

with eight 1250-watt heaters placed in the heating section at 6-inch intervals along the vertical axis. Additional heaters were provided on the gas transfer line to reduce the heating time.

The condensers were simple tube-in-shell heat exchangers constructed from 1-1/2-inch and 3/4-inch deoxidized copper pipe with headers for the oil receivers. The condensers were designed and constructed to operate counter-currently using return gas from the brine cooled condenser as the cooling medium in the first two condensers, water in the third condenser, and brine in the fourth condenser.

A gas circulation section consisting of a gas pump and appropriate valves was constructed to circulate the gas through the system. Temperature control and measuring equipment was provided for control of the gas temperature to the retort, and for measuring the temperature of the entering and exit material in each section.

On retorting 16-1/4 pounds of oil shale for one hour at a gas pressure of 10 pounds per square inch gage, yields of 50.5, 47.3, 40.9, and 39.4 per cent of the available shale oil were obtained from oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), respectively.

The A.P.I. gravity was found to increase from 15.2, 17.9, 18.6, and 22.4 degrees, and the kinematic viscosity

decrease from 12.18, 4.64, 2.94, and 1.66 centipoises for a decrease in particle size from minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale), respectively. The mid-boiling point of the shale oil decreased 510, 500, 482, and 380 °F for the above particle sizes as did the average boiling point which decreased from 824, 724, 690, and 590 for oil shale of the same particle size.

The yield of non-condensable gases increased 0.67, 0.72, 1.01, and 1.2 cubic feet for oil shale of minus 16 plus 28, minus 28 plus 70, minus 70 plus 100, and minus 100 plus 200 mesh (Tyler screen scale).

A decrease in particle size of oil shale retorted at 715 °F in a 4-inch fluidized retort using carbon dioxide as the fluidizing medium, was found to definitely reduce the mid-boiling point, average boiling point, and the kinematic viscosity of the shale oil, and to increase the A.P.I. gravity of the shale oil obtained as well as the volume of retort gas.

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