STATE OF COLORADO
COOPERATIVE OIL SHALE INVESTIGATION
(In Cooperation With the United States Bureau of Mines)

MARTIN J. GAVIN, Engineer in Charge

BULLETIN NO. 1
Boulder, July 1, 1921

Short Papers from the Cooperative Oil-Shale Laboratory

By

MARTIN J. GAVIN and LESLIE H. SHARP

Denver, Colorado
EAMES BROTHERS, STATE PRINTERS
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LETTER OF TRANSMITTAL

DEPARTMENT OF THE INTERIOR
BUREAU OF MINES
WASHINGTON

TO HIS EXCELLENCY,
The Honorable Oliver H. Shoup,
Governor of Colorado.

Sir:

I have the honor to transmit six short papers by M. J. Gavin, Oil Shale Technologist, U. S. Bureau of Mines, and L. H. Sharp, Chemical Engineer for the State of Colorado. These papers present the results of certain studies made at the Co-operative Oil Shale Laboratory, Boulder, Colorado. Further reports of the studies, which are still in progress, will be transmitted when completed.

Cordially yours,

H. FOSTER BAIN,
Director,
U. S. Bureau of Mines.

PREFACE.

This paper is a presentation of the results of preliminary studies at the Colorado Co-operative Oil Shale Laboratory, Boulder, Colorado, which were begun February 1, 1920, by the U. S. Bureau of Mines and the State of Colorado, under a co-operative agreement entered into by the Bureau and the State, utilizing funds which were provided by the State, and the services of engineers provided by the Bureau.

The investigations are for the purpose of determining by large-scale laboratory retorting tests, those conditions which will produce optimum yield of best quality of products from Colorado shales.

It seems fairly certain that the peak of the petroleum production curve in the United States will be reached in a few years, but the curve of consumption will contine in its upward rise. To meet this situation, either imports must be increased in the future, or means must be found to utilize the immense oil-shale deposits of Colorado and other western states as a source of the needed oil.

This last is not a simple problem. While oil shales have been worked in Scotland and France for many years, it was in competition with high-priced petroleum products and with low labor costs, with the added advantage that the industry is there situated in a densely populated region where a ready market for oil and ammonimum products was available. Even these long-established industries are passing through a difficult period at present. The oil shales of the Rocky Mountain region occur in sparsely settled communities and their development will mean bringing into the region great numbers of working men, with their families, for whom housing and the conveniences of living must be provided, in addition to the millions of dollars which must be spent in constructing plants, equipping mines, and providing transportation facilities. About one million barrels of oil are now produced each day in the United States and to produce one barrel of oil from oil-shale will involve the mining and crushing of at least one ton of tough material, heating it to a high temperature and finally disposing of three-fourths of a ton of waste residue.

It naturally follows that an enterprise which bids fair to be so important to the State of Colorado justifies the most careful investigation to assure that development shall be along the right lines, since the loss of capital resulting from too-hasty construction of unsuitable plants would be certain to prove an obstacle to securing the needed capital for the development of the industry. Until the fundamental factors underlying the development of the oil shales of the Rocky Mountain region have been clearly and accurately ascertained, no sound development of the oil shale industry will be possible.

A. W. AMBROSE,
Chief Petroleum Technologist,
U. S. Bureau of Mines.

Washington, D. C.,
May 15, 1921.

Plate II. Typical Oil-Shale Ledge in Colorado

Plate III. Typical Oil-Shale Formation in Colorado

INTRODUCTION.

In January, 1920, the State of Colorado and the United States Bureau of Mines entered into a co-operative agreement for the conduct of laboratory investigations on the oil-shales of Colorado. Under this agreement a laboratory has been installed and equipped at the State University, Boulder, Colorado, and a research staff organized. It is the primary purpose of the investigational work to determine the most favorable conditions of retorting Colorado oil-shales to yield the most of the best products from them.

Work of this nature involves retorting the oil-shale under many conditions and the examination of products obtained in each test to determine the effect of the conditions imposed during the test. New problems continually arise which call for the development of new methods for their solution and frequently interesting developments are investigated only to give results of negative value. It becomes apparent that a great deal of time will be required before the main purpose of the investigation can be accomplished. However, in the course of the main investigation it was necessary to take up certain side investigations which were directly connected with the main plan of the work. Many of these minor investigations have yielded results of sufficient interest and importance that it has been considered worth while to bring them to the attention of the public before the principal results of the main investigation can be published.

Two papers[1] dealing with the program of the investigations and with some of the work already accomplished have already been published in mimeographed form by the Bureau of Mines, and it is the purpose of the Bureau and State to continue publishing short reports as frequently as material becomes available. The final results of the completed investigations are to be the subject of a Bureau of Mines bulletin.

This present paper deals with several subjects which will be of interest to those engaged in the development of an industry from the immense deposits of oil-shales in Colorado and adjacent states. It is a compilation of six short reports which have been prepared in the course of the investigational work. The fuel values of oil-shale and oil-shale products are discussed in the first paper; the nature and composition of shale gas is presented in the second; the third gives production tables and curves for shale oil as obtained from the horizontal rotary retort used in the Boulder laboratory; the analytical distillation of shale oils is taken up in the fourth report; the fifth gives data on thermal calculations for the retorting of oil-shales, and the sixth is a tabulation of factors and formulae which have been found of value in the Boulder co-operative laboratory and the oil-shale laboratory at the Intermountain Experiment Station of the Bureau of Mines, Salt Lake City, Utah.

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[1] Gavin, M. J., and Sharp, L. H., Investigation of the fundamentals of oil-shale retorting, Bureau of Mines. Reports of Investigations, Serial No. 2141, July, 1920, 4 pp. Reprinted in Eng. World, Sept., 1920.

Gavin, M. J., and Sharp, L. H., Some physical and chemical data on Colorado oil-shale, Bureau of Mines, Reports of Investigations, Serial No. 2152, August, 1920, 8 pp. Reprinted in Eng. and Min. Jour., Sept. 18, 1920; Oil Paint and Drug Reporter, Sept. 13, 1920; and Gas Age, Sept. 25, 1920.

Attention is called to the fact that the data given, except those in the last paper, can be expected to apply only to the oil-shale worked with and the products recovered therefrom *under the particular conditions* used in the investigations. However, the material being worked with is believed to be a fairly representative sample of Colorado oil-shale, and if due allowances are made for the varying richness of different shales, the results may be expected to be applicable, with a reasonable degree of accuracy, to all shales of the Green River formation.

ACKNOWLEDGEMENTS.

The writers gratefully acknowledge the services rendered by Mr. James Duce, State Oil Inspector of Colorado, in perfecting co-operative agreements and in arranging for laboratory and office space, and are especially grateful to him for the many valuable suggestions he has made and for the deep interest he has taken in the work.

To Professors John A. Hunter and Jay W. Woodrow as well as other faculty members and the regents of the University of Colorado, thanks are due for the co-operative spirit shown by them and for the material assistance they have rendered in many ways. Acknowledgements are made to Alvah M. Hovlid, of the Co-operative Laboratory, Boulder, for assistance in carrying out much of the experimental work leading to the results herein presented, and to L. C. Karrick and J. J. Jakowsky, the authors' associates in the Bureau of Mines Experiment Station, Salt Lake City, Utah, for assistance in preparing manuscript and for valuable suggestions as to the conduct of the experimental work. Mr. Jakowsky also prepared curves Nos. 1 to 9. Manuscript was prepared by Miss Louise Helson of the Salt Lake City Station of the Bureau of Mines, and Mr. A. T. Strunk of the Boulder Laboratory. Mr. Arthur J. Franks of Golden, Colorado, kindly supplied certain results of his oil-shale studies for use in connection with the paper on Thermal Calculations on the Retorting of Oil Shales. The manuscript was constructively critised by T. E. Swigart and N. A. C. Smith of the Bureau of Mines.

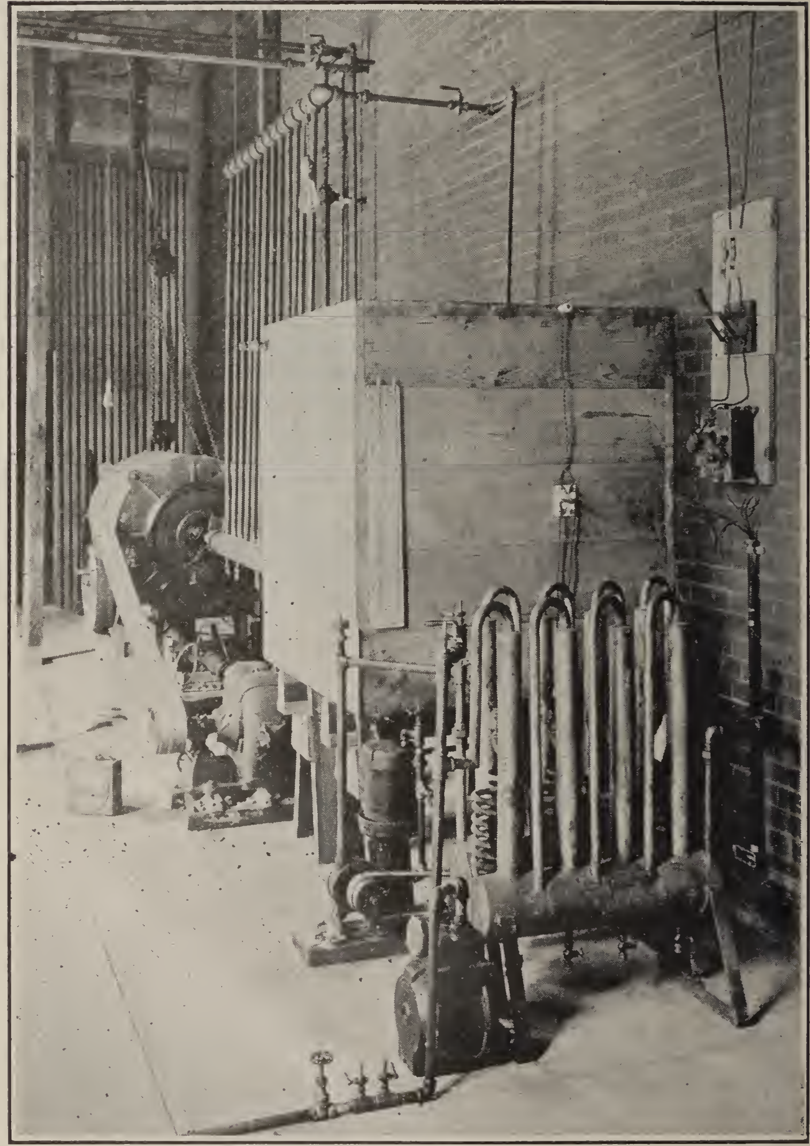

Plate IV. Horizontal Retort at Boulder Laboratory. Scrubbers in Foreground

FUEL VALUES OF OIL-SHALES AND OIL-SHALE PRODUCTS.

Subsequent to the publication[1] of the heats of combustion of a fresh oil-shale yielding 42.7 gallons of oil to the ton, and the spent shale and oil derived therefrom, it has seemed advisable to secure similar data for shales of varying oil yield and different physical and chemical characteristics, in order to furnish geologists and engineers with an accurate basis for calculating the thermal values of oil-shales and their products, especially for possible retort fuels.

Accordingly, six samples of shale of diversified character as indicated below were selected, determinations of the heats of combustion of the fresh shales and of the spent shales and oils derived from them were made, and the heat value of the derived gases calculated.

CHARACTERISTICS OF SHALES USED.

No.	Physical Composition	Chemical Nature	Oil Yield on Assay; Gals. Per Ton	From Locality	Nature Oil	Water Yield on Assay Gals. Per Ton
1	Massive	Limy	10.0	Dry Fork[1]	Light	1.06
2	Massive	Siliceous	28.0	Conn Creek[1]	Med. waxy	4.22
3	Massive	Siliceous	37.0	Conn Creek[1]	Med. waxy	3.16
4	Massive	Siliceous	42.7	Conn Creek[1]	Med. waxy	3.16
5	Thin "paper"	Mouldy organic	75.5	Dry Fork[1]	Gassy light	6.04
6	Semi-massive "paper"	Siliceous organic	76.2	Mt. Logan[1]	Med. waxy	6.30

[1] Near DeBeque, Colo.

In all cases the samples were crushed to —¼ mesh in a chipmunk crusher, the crushed shale thoroughly mixed and sampled, then approximately one pint of each shale was taken for assay.

The assays were made by the method recommended by the U. S. Bureau of Mines for oil-shale assay.[2] The residues were carefully weighed and sampled and distillation losses noted. Oils were preserved in glass stoppered flasks. All samples of fresh and spent shale were ground to pass a 100 mesh screen and heats of combustion determined in the standard Emerson bomb calorimeter. The heat of combustion of oils was then determined in the same apparatus. (300 to 350 pounds of oxygen pressure were used in all determinations and temperature readings were taken with a Beckman differential thermometer.) Check determinations were run.

The determinations were corrected for unburned material[3] as shown in the following table. A further check in the form of total ignition loss determination was made.

---

[1] Gavin, M. J., and Sharp, L. H., Some physical and chemical data on Colorado oil-shale, Bureau of Mines, Reports of Investigations, Serial No. 2152. August, 1920. 8 pp.

[2] Karrick, L. C., A convenient and reliable retort for assaying oil-shales for oil yield, Bureau of Mines, Reports of Investigations, Serial No. 2229, March, 1921. Reprinted in Eng. and Min. Jour., April 30, 1921.

[3] Gavin, M. J., and Sharp, L. H., Some physical and chemical data on Colorado oil-shale, Bureau of Mines, Reports of Investigations, Serial No. 2152, August, 1920. 8 pp.

TABLE I.

SUMMARY—HEATS OF COMBUSTION OR FUEL VALUES OF OIL-SHALE AND ITS PRODUCTS.

Sample No. [1]	1	2	3	4	5	6	Wyoming Shales 7	 8	 9
Specific gravity of shale	2.29	2.09	2.13	2.00	1.51	1.56			
Oil yield, gallons per ton[2]	10.0	28.0	37.0	42.7	75.5	76.2	39.4	39.1	30.4
Specific gravity oil	0.880	0.913	0.919	0.917	0.888	0.908	0.939	0.937	0.897
Heat of combustion oil:									
Calories per gram	10400	10194	10400	10215	10142	10495			
B. T. U. per pound	18710	18349	18709	18387	18255	18871	(average 1-6: 18547)		
Water yield, gallons per ton[2]	1.06	4.22	3.16	3.16	6.04	6.30	1.85	1.58	2.64
Loss on distillation, per cent	8.15	16.10	20.80	22.90	37.70	39.80	31.2	31.4	26.9
Loss on ignition, per cent	36.40	37.30	42.80	40.70	54.05	55.25	54.6	52.5	49.2
Heat of combustion shale:									
Calories per gram	573	1744	2250	2460	3845	4430			
B. T. U. per pound	1031	3139	4050	4428	6921	7974			
$F=\frac{\text{Heat combustion shale, B. T. U. per lb.}}{\text{Gallons oil per ton}}$	103.1	112.3	109.4	103.3	91.8	104.8	(Average 103.9; average excluding No. 5, paper shale, 106.6.)		
Heat of combustion spent shale[3]:									
Calories per gram	136	452	473	600	1024	924			
B. T. U. per pound	245	814	851	1080	1845	1663			
Per cent of heat value in gram of spent shale[4]	23.75	25.9	21.0	24.40	26.60	20.9	(Average 23.76; average excluding No. 5, 23.19)		

---

[1] Numbers refer to samples described on page 13.
[2] On laboratory assay.
[3] Spent shale is residue from dry distillation in assay retorts.
[4] Heat of combustion per gram of spent shale divided by heat of combustion per gram of fresh shale.

A careful examination of this table for various ratios and relationships yields the results noted below.

A. It is often said that the lighter the shale the higher its oil yield. Accordingly the *oil yield* was divided by the reciprocal of the *specific gravity* but the result was far from constant. Various other attempts to find a mathematical relationship failed. So far as these experiments indicate, there is none.

B. The *distillation loss* of oil shale is composed of: *(a)* the oil volatilized, *(b)* the water volatilized, *(c)* the gas formed, and *(d)* variable positive and negative losses due to decomposition of some of the chemical constituents of the mineral matter and oxidation of others.

The percentage of loss due to each of the above is as follows:

(a) Number of gallons of oil per ton of shale × 0.375. (specific gravity = 0.900). (Factor obtained by dividing weight of a gallon of shale oil by 2000 (pounds in a ton) × 100.)

(b) Gallons of water per ton of shale × 0.416. (Factor obtained by dividing weight of a gallon of water by 2000 × 100.)

(c) Cubic feet of gas per ton of shale × 0.00025. (density = 0.656; air = 1.000). (Factor obtained by dividing weight of a cubic foot of gas by 2000 × 100.)

(d) Sum of $(a + b + c)$ subtracted from total per cent loss on distillation as shown by assay or plant records.

C. In a very general way it may be said that the greater the *ignition loss* the greater the *oil yield.* This relation, however, must not be accepted as a satisfactory basis for estimating oil yields from oil-shales, because ignition losses include losses that do not go to make up oil, on distillation, such as losses due to decomposition of the carbonates in the shale, water of crystallization, and the like.

D. When the *oil yield is* compared with the *heat of combustion* a somewhat more definite ratio is found. Dividing heat of combustion of the shale in B. T. U. per pound by assay yield of oil in gallons per ton, gives a series of numbers averaging 103.9, and omitting the "paper" shale[1], averaging 106.6, with a variation between samples of less than ± 5.0 per cent. This number is the factor F in Table I.

Conversely, if the assay yield of oil in gallons per ton of any shale is known, its heat value in B. T. U. per pound can be closely approximated by multiplying oil yield by this factor (106.6). If it is desired to express the heat value of the shale in calories per gram, the above factor becomes 59.24.

Example: An oil-shale is assayed and found to yield 50 gallons of oil per ton.

$$\left.\begin{array}{l}50 \times 106.6 = 5330 \text{ B. T. U. per pound} \\ \text{or } 50 \times 59.24 = 2960 \text{ calories per gram}\end{array}\right\} \text{Heat of combustion of shale.}$$

---

[1] All the results obtained seem to indicate a different set of constants for "paper" shales.

Later calculations *(not experimental evidence)* seem to indicate that the fuel value factor may decrease slightly for the shales yielding much over 60 gallons of oil per ton. The factor given, however, is sufficiently close to serve as a good approximation for most shales, especially as shales yielding over 50 gallons of oil per ton are rather exceptional.

The available heating value of an oil-shale will, of course, be influenced by the water content of the shale, since the water must be vaporized during combustion, when it is present, and thus subtracts from the total heating value as calculated by the above method.

It may be that the composition of kerogen in the shales of various widely separated localities or geological horizons may be sufficiently diverse to necessitate separate determinations of the above factor. The problem merits further investigation. The variation of the "paper" shale considered in connection with the character of oil yielded by it, argues for a different factor in this case at least.

E. The *heat of combustion* of the *spent shale* in these tests varies from 12.58 to 21.85 per cent of that of the raw shale from which it is derived, and averages 17.42 per cent of it. This factor was determined by multiplying weight of spent shale from one gram of raw shale by the heat of combustion of the spent shale. (See Table IV.)

Example: Heating value of one ton of oil-shale yielding 50 gallons of oil per ton = 10,660,000 B. T. U.

10,660,000 B. T. U. × 0.1742 = 1,858,000 B. T. U. (Fuel value of spent shale from one ton of raw 50-gallon shale.)

F. The heat of combustion of the oil recovered from the shales examined varied from 62.45 to 74.40 per cent of the heat value of the shale from which it was recovered, and averaged 67.06 per cent of it. Tables III and IV indicate how this factor varies and how it was derived.

Example: Heating value of one ton of 50-gallon oil-shale = 10,660,000 B. T. U.

10,660,000 × 0.6706 = 7,148,000 B. T. U. (Heating value of oil obtained from one ton of this shale.)

G. The heat of combustion of the gas obtained from the shales used in these tests varied from 9.00 to 18.10 per cent of the heat value of the shale from which it was obtained, and averaged 15.52 per cent of it. Table IV also indicates how this factor varies among the different samples examined.

Example: Heating value of one ton of 50-gallon oil-shale = 10,660,000 B. T. U.

10,660,000 × 0.1552 = 1,654,000 B. T. U. (Heating value of gas obtained from one ton of this shale.)

In none of the tests reported in this paper was gas production forced to its limit. If the shales had been heated to a higher temperature, or held at the maximum temperature reached for a longer time, a greater quantity of gas would have been recovered, and the heating value of the gas produced would be a greater percentage of the heat value of the raw shale than is indicated above. Such gain by the gas would be at the expense of the spent shale, whose weight and total heating value would become less as gas production reached a maximum.

Considering the diversity of the samples tested as to physical and chemical nature, oil yield, and geologic position, it is reasonable to believe that the different factors developed above will be of very general application. It should be noted again, however, that some of them may not be applicable to paper shales and shales similar to them.

Below are given Tables II, III and IV, all of which have been developed from material presented in Table I and the above discussion. Following these is Table V which gives data on fuel values of different fuels for use in making comparisons. Table II presents heat values necessary for use in calculating heat balances; Table III shows the actual distribution of the heat value of the raw shale among its combustible products; and in Table IV the percentages of the total heat values of the shales examined, appearing in spent shale, oil and gas, are given as well as average values.

TABLE II.

FACTORS NECESSARY FOR CALCULATING HEAT BALANCES.

Sample No.	1	2	3	4	5	6
Weight fresh shale (grams)	1	1	1	1	1	1
Distillation loss, per cent	8.15	16.10	20.80	22.90	37.70	39.80
Weight spent shale (grams)	0.918	0.839	0.792	0.771	0.623	0.602
Heat of combustion spent shale, Calories per gram[1]	[2]136	452	473	600	1024	924
Heat combustion spent shale from 1 gram shale, calories	122	380	374	463	638	557
Oil yield (cc. per lb.)	19.0	53.2	70.0	81.0	143.0	144.5
Old yield (cc. per gram)	0.0418	0.117	0.154	0.178	0.315	0.518
Specific gravity oil at 15.5° C.	0.880	0.913	0.919	0.917	0.888	0.908
Oil yield (gram per gram)	0.0368	0.107	0.1415	0.163	0.282	0.289
Heat combustion oil, calories per gram[1]	10400	10914	10400	10200	10142	10495
Heat of combustion oil from one gram of shale, calories	383	1090	1470	1661	2860	3180

[1] To change calories per gram to B. T. U. per pound, multiply by 1.8.

[2] Calculated from average per cent heat value in spent shale (see Table I).

TABLE III.

HEAT DISTRIBUTION.

In One Gram Fresh Shale and Products Obtained from It.

Sample No.	1	2	3	4	5	6
Heat combustion fresh shale, calories	573	1744	2250	2460	3845	4430
Heat combustion spent shale, calories[1]	[2]122	380	374	463	638	557
Heat combustion oil	383	1090	1470	1661	2860	3180
Heat combustion gas, by difference (calories)[3]	68	274	406	336	347	693
Sum of heat values of products, calories	573	1744	2250	2460	3845	4430

[1] Weight of spent shale × its thermal value = gram calories in spent shale.

[2] Calculated from averages.

[3] See pages 16 and 19.

TABLE IV.

PERCENTAGE HEAT DISTRIBUTION.

Sample No.	1	2	3	4	5	6	Av.	Average excluding No. 5
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	
Spent shale	[1]17.29	21.85	16.60	18.80	16.60	12.58	17.29	17.42
Shale oil	68.28	62.45	65.30	67.50	74.40	71.77	68.28	67.06
Shale gas[2]	14.43	15.70	18.10	13.70	9.00	15.65	14.43	15.52
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

[1] Calculated from corresponding averages this table.
[2] By difference.

TABLE V.

HEATING VALUES OF SHALE AND SHALE PRODUCTS COMPARED WITH OTHER FUELS.

	Heat of combustion Cal. per gm.	Heat of combustion B. T. U. per lb.	No. tons needed to equal 1 ton lignite 6800 B.T.U. heating value	No. tons needed to equal 1 ton bitum. coal of 12500 B. T. U. heating value
Solid:				
Fresh shale, 25 gal. oil per ton	1482	2665	2.55	4.69
Fresh shale, 50 gal. oil per ton	2962	5330	1.28	2.34
Fresh shale, 75 gal. oil per ton	4440	7995	0.851	1.56
Fresh shale, 100 gal. oil per ton	5935	10660	0.638	1.17
Spent shale, 25 gal. oil per ton	352	633	10.73	19.75
Spent shale, 50 gal. oil per ton	705	1267	5.36	9.87
Spent shale, 75 gal. oil per ton	1055	1900	3.58	6.58
Spent shale, 100 gal. oil per ton	1409	2535	2.68	4.93
Lignite	3526-3994	6347-7189		
Peat (air dried)	4867-5726	8761-10307		
Coal, bituminous	6088-7852	10958-14134		
Coal, anthracite	6987-7417	12577-13351		
Coke	7000	12600		
Liquid:				
Shale oil, sp. gr. 0.917	10215	18387		
Shale oil, sp. gr. 0.880	10400	18709		
Fuel oil, sp. gr. 0.903	10710	19280		
Fuel oil, sp. gr. 0.880	10790	19410		
Fuel oil, sp. gr. 0.853	10905	19610		
Gaseous:		per cu. ft.		
Shale gas, early stages[1] ... A		482.0		
Shale gas, oil 15 to 90 per cent off[1] ... B		976.0		
Shale gas, oil 90 to 100 per cent off[1] ... C		526.0		
Shale gas, oil all off[1] ... D		213.0		

	per cu. ft.	No. cu. ft. shale gas to equal 1 cu. ft. other gas A	B	C	D
Natural gas[2]	1000	2.08	1.03	1.90	4.70
Oil gas[2]	634	1.32	0.65	1.21	2.98
Coal gas[2]	683	1.42	0.70	1.30	3.20
Producer gas[2]	153	0.32	0.16	0.29	0.72
"Blue" water gas[2]	322	0.67	0.33	0.61	1.51

[1] Obtained by dry destructive distillation in batch retort; efficiency of gas scrubbing doubtful.
[2] Average values.

In the discussion under paragraphs E, F, and G, certain factors were presented by means of which, if the heat value of a sample of shale is known, the heating value of its products—gas, oil, and spent shale—can be calculated. These factors represent the percentage of the total heating value of the raw shale appearing in each of the products, and as can be noted in Table IV, the percentage of the original heating value of the shale found in the gas is determined by difference from 100 per cent, as the heating values of the shale, oil, and spent shale have been experimentally determined. On first impression, it would appear that the sum of the heat values of the products of oil-shale should equal the heat value of the raw shale, but this does not necessarily follow.

As a matter of fact the percentage distribution of heating values, as shown in Table IV, applies very well for shales yielding up to 50 gallons of oil per ton, but for richer shales experiments in which the actual heating values of shale gas were determined, have indicated that the percentages representing heating value distribution must be somewhat modified to obtain fuel values for the spent shale and shale gas that are consistent with actually observed values. It has been determined by experience that the following distribution of the heating value of the raw shale among its products agrees closely with observed values for shales of different richness:

(If the total heat value of the raw shale is found from the formula: 106.6 × oil yield in gallons per ton = B.T.U. per pound of shale.) (See page 16.)

For shales yielding	Up to 50 gals. oil per ton	50 to 80 gals. oil per ton	80 to 100 gals. oil per ton
Percentage of total heating value of raw shale found—			
In oil	65.00	65.00	65.00
In spent shale	18.65	15.00	11.00
In gas	15.35	16.00	16.00
Percentage unaccounted for	1.00	4.00	8.00

The figures shown in the first column are rounded averages for those shales discussed in this paper which yielded up to 50 gallons of oil per ton, and it is believed that they may be applied without serious error. The values given in the other columns have been somewhat arbitrarily chosen from results on rich shales not reported in this paper. As most shales which will be commercially worked do not usually yield over 50 gallons of oil to the ton, it was not thought worth while to spend any great amount of time in determining factors for richer shales.

It is interesting to consider what becomes of that part of the heating value of the raw shale designated as "unaccounted for" when the shales are distilled. It is entirely possible that there have been high distillation losses in the distillation of the richer shales, which may not have been observed, but the writers do not believe this to be the case. The decomposition of oil-shale into its products is a thermo-chemical process, and it seems most likely that the heat unaccounted for represents, in a measure at least, the heat of reaction of the distillation process. The heat of reaction of the process undoubtedly differs with different shales, and with the same shales when they are distilled under different thermal conditions, thereby producing different end products.

SUMMARY.

The results of the work presented in this paper make it possible to draw the following conclusions:

1. There is no mathematical relationship between the specific gravity of an oil-shale and the amount of oil yielded by it. The idea that shales of low specific gravity yield much oil, if used at all, must be applied with caution.

2. The ignition loss of an oil-shale cannot properly be used in estimating the amount of oil the shale will yield.

3. The heat of combustion of an oil-shale is a fairly accurate indicator of the amount of oil the shale will yield, and, conversely, the oil yield is a reliable indicator of the heat value of the shale as a fuel.

4. The heat value per gram of spent shales apparently tends to approximate a constant percentage of the heat value per gram of the shales from which they were formed, rather than a constant average heat value, when the shales are retorted under constant conditions. For the conditions of retorting used in these experiments, this percentage is 23.19.

5. In the experiments reported in this paper, the amount of heat recoverable in the shale oil tends to approximate a definite percentage of the heat of combustion of the original shale. For the conditions of retorting used in these experiments this percentage is 68.28.

If, as is here indicated, only 68.28 per cent of the original fuel value of the shale is contained in the oil recovered by dry destructive distillation, it seems highly desirable, from a viewpoint of national economy, that both the spent shale and shale gas be used as fuel to the fullest extent. When the fuel values of these latter products are considered, however, it is doubtful if such use will always be the most profitable from a financial standpoint.

6. For fairly approximate work it can be taken that the oil yield of a shale (as determined by assay) in gallons per ton, multiplied by the factor 106.6 equals the gross heat of combustion of the shale in B.T.U. per pound.

7. To obtain the *net heat value* the factor 106.6 may be corrected as follows:

> *(a)* For every gallon of water per ton which is vaporized, subtract 4.66 from the 106.6 factor.
>
> *(b)* For every degree Fahrenheit above 212° F. (boiling point water) each gallon of vaporized water (steam) is raised in temperature before its discharge, a further subtraction of 0.002 should be made from the 106.6 factor.
>
> *Example:* A shale assaying 50 gallons oil and 2 gallons water per ton, is used as fuel where flue gas exit temperature is 612° F.

Gross heating value is 50 × 106.6 = 5330 B.T.U. per pound. Net heating value is 50 × [106.6 — (2 × 4.66) — (400 × 0.002)] = 50 × 96.48 = 4820 B.T.U. per pound.

8. For shales yielding up to 50 gallons of oil per ton the following relations can be used for close approximations:

(a) Heat value of raw shale multiplied by 0.1742 equals the total heat value in B.T.U. of the spent shale derived from it.

(b) Heat value of raw shale multiplied by 0.6706 equals the total heat value in B.T.U. of the oil derived from it.

(c) Heat value of raw shale multiplied by 0.1552 equals the total heat value in B.T.U. of the gas produced from it.

These relationships hold only for dry destructive distillations under the conditions used in these tests. For shales richer than those yielding 50 gallons of oil to the ton, the above factors must be modified as indicated on page 19.

OBSERVATIONS ON SHALE GAS.

Frequent mention has been made of the possibility of supplying all or part of the heat necessary to retort oil-shale by burning the uncondensible gas under the retort as fuel. This paper has been prepared to present the findings with regard to the feasibility of this plan.

In a paper on the "Fuel Values of Oil-Shales and Oil-Shale Products" (see Table IV) the writers show that from 9.0 to 18.1 per cent of the heat value of the shale is represented, after retorting by dry destructive distillation, by the uncondensible gases. The average for the shales examined was about 15.0 per cent. From this it is evident that the total heat value obtainable from uncondensible gases varies much with different shales. It will also differ with different conditions of retorting.

The tests described below show that the heating value of the gas also varies more or less according to time at which the gas is formed with reference to oil production. This statement applies also to the chemical composition of the gas.[1]

In Table VI the results tabulated under Shale No. 10 are the average of those obtained in four retort tests on 75-pound samples of shale assaying 42.7 gallons of oil per ton; those under Shale No. 11 are an average of observations made on four retort tests using 75 pounds of shale yielding, on assay, 37 gallons of oil per ton; and those under Shale No. 12 are the observations made on a single retort test using 75 pounds of shale yielding, on assay, 28 gallons of oil per ton.

---

[1] During tests on shale samples Nos. 11 and 12, Table VI, all gas was scrubbed, an average of 0.115 gallons of gasoline being absorbed from 1000.0 cubic feet, as follows:

Test No. 5. 36.75 cc. from 132.8 cu. ft. or 0.0728 gals. per 1000 cu. ft.
Test No. 6. 23.00 cc. from 36.3 cu. ft. or 0.167 gals. per 1000 cu. ft.
Test No. 7. 46.00 cc. from 81.5 cu. ft. or 0.149 gals. per 1000 cu. ft.
Test No. 8. 26.00 cc. from 93.5 cu. ft. or 0.0733 gals. per 1000 cu. ft.

There is a possibility that the scrubbing was somewhat incomplete on account of a too rapid gas flow.

The gas samples referred to were produced in the course of shale retorting tests made in the United States Bureau of Mines and State of Colorado co-operative oil-shale retort at Boulder, Colorado. Briefly, this retort is an externally gas fired, horizontal, rotary, iron cylinder with a pyrometer well in one end and the vapor exit in the other. The vapors are drawn through an air-cooled and a water-cooled condenser, then pumped through water and a light "straw" oil. On leaving the oil scrubber the gases are metered. The heating value of the gas is next determined with a Junkers calorimeter set. Samples are collected over water for analysis by a standard portable Williams Orsat pipette.

TABLE VI.

TABLE SHOWING RELATION BETWEEN GAS PRODUCTION, OIL PRODUCTION, TEMPERATURE AND HEATING VALUE OF GAS PRODUCED.[1]

	Shale No.[2]	Heating value gas B. T. U. per cu. ft.	Per cent oil off	Retort temp. at time of taking gas sample; deg. F.[3]	Rate of formation		Av. temp. rise of retort before taking gas sample; deg. F. per min.[3]	Rate retort temp. rise for test deg. F. per min.[3]
					Gas cu. ft. per min.[4]	Oil cc. per min.[4]		
Before 15 per cent oil is distilled	10	482	14.0	420	0.8	35.3	[5] 4.66	4.56
After 15 per cent and before 90 per cent oil is distilled	10	1043	27.45	560	0.333	34.65	......	4.56
After 15 per cent and before 90 per cent oil is distilled	11	891	48.88	586	0.280	65.56	2.42	3.55
After 15 per cent and before 90 per cent oil is distilled	11b[6]	994	47.78	453	0.283	62.63	1.74	3.55
After 15 per cent and before 90 per cent oil is distilled	12							
After 15 per cent and before 90 per cent oil is distilled	Av.	976	41.37	533	0.299	54.28	2.09	......
After 90 per cent and before 100 per cent oil is distilled	10	465	95.18	775	0.300	27.67	3.0	4.56
After 90 per cent and before 100 per cent oil is distilled	11	800	96.66	810	0.530	38.67	3.78	3.55
After 90 per cent and before 100 per cent oil is distilled	12	312	97.10	865	undet.	5.40	3.67	6.38
After 90 per cent and before 100 per cent oil is distilled	Av.	526	96.31	817	0.415	23.91	3.48	......
After all oil is distilled	11	213	100.00	1158	0.200	....	3.33	3.55

---

[1] Volume of gas indicated is that observed at 75° F. and 622 mm. barometric pressure. The B. T. U. observations refer to gas reduced to 0° C. and 760 mm. pressure.

[2] Shale No. 10, oil yield 42.7 gals. per ton. Figures average of tests Nos. 1, 3, 4.
Shale No. 11, oil yield 37.0 gals. per ton. Figures average of tests Nos. 5, 6, 7, 8.
Shale No. 12, oil yield 29.0 gals. per ton. Figures of test No. 9.

[3] Temperature as indicated by pyrometer located coaxially in rotating retort cylinder; does not necessarily indicate temperature of shale.

[4] Gas production given in cubic feet per minute from 75 pounds shale; oil production given in cubic centimeters per minute from 75 pounds shale.

[5] The figures in this column indicate the rate of temperature rise in degrees Fahr. per minute for the 15-minute period before the sample was taken.

[6] 11b omits one anomalous B. T. U. determination of 274 from average.

CONCLUSIONS.

The following conclusions may be reached as a result of the experimental evidence presented in Table VI and later work, results of which were unavailable for presentation in detail in this bulletin.

1. In general the thermal value of the gas rises with the temperature at which the gas was formed, until some 90 per cent of the oil obtainable has been distilled from the shale.

2. Before 15 per cent of the obtainable oil is distilled from the shale, the heat value of the gas is approximately 482 B.T.U. per cubic foot.

3. After 15 per cent of the possible oil has been recovered, and until 90 per cent is obtained, the thermal value of the gas rises to an average of 976 B.T.U. per cubic foot.

4. After 90 per cent of the obtainable oil has been recovered, the average thermal value of the gas is about 526 B.T.U. per cubic foot, or very similar to that obtained during the time of producing the first 15 per cent of the oil.

5. After all the oil has been yielded by the shale, the thermal value of the gases formed drops to a value of about 213 B.T.U. per cubic foot, and probably remains between 200 and 300 B.T.U. until gases cease to be evolved.

6. In the early stages of retorting there seems to be no definite relation between the thermal value of the gas and the rate of oil production. This seems to hold true until after 90 per cent of the oil yield is obtained, after which time the heating value of the gas seems to fall off, roughly as the rate of oil production decreases.

7. There is apparently no connection between the rate of gas production and its heating value, or between the rate of temperature rise just before the calorific determination, and the value of the latter.

It was intended that a gas sample, for analysis, should be collected during or immediately after each calorific value test. The samples were collected but due to breakage of apparatus it was necessary to delay some of the analyses until their results were manifestly incorrect, and therefore only three are submitted. These three are results obtained on freshly collected samples and are therefore believed to actually represent the gases as they were produced.

The exact conditions of the tests, so far as the apparatus permitted their observation at the time of sampling the gases, together with the analyses of the gases, are shown in Table VII. Average analyses of various other natural and artificial gases are also included in the table for comparison.

The authors appreciate that it is unjustifiable to draw conclusions from the results of so few analyses. The analyses are appended, however, to show the nature of the work under way and to give, at least, a preliminary idea of the nature of the shale gas obtained under the conditions prevailing in the experimental work.

TABLE VII.

COMPOSITION OF SHALE AND OTHER GASES.[1]

	Shale gas No. 1	Shale gas No. 2	Shale gas No. 3	Coal gas, Manchester, 1857	"Blue" water gas	Carburetted water gas, Anthracite	Oil gas, San Francisco	Natural gas, Pennsylvania	Pintsch gas	Producer gas (Mond)	Scotch shale gas
CO_2, per cent	9.6	4.5	17.0	3.67	4.60	4.50	2.62	0.26	0.20	13.9	20-30
CO, per cent	2.4	1.6	8.4	6.64	39.75	29.00	9.21	0.50	0.50	13.8	2-5
Ill (CnH_2n), per cent	9.0	6.9	11.5	6.46	0.00	13.00	7.01	0.31	38.10	0.0	tr.-2.0 (Max. 6%)
CH_4, per cent	56.1	32.6	29.4	34.90	2.20	16.00	34.64	92.60	57.70	2.0	7-10
H_2, per cent	0.0	8.2	15.7	45.58	50.00	32.00	39.78	2.18	3.40	24.3	25-60 (Av. 41%)
O_2, per cent	4.0	7.9	3.1	0.00	0.35	0.50	0.16	0.34	0.00	0.0	Max. 4%
N_2, per cent	18.9	38.3	14.9	2.75	3.10	5.00	6.58	3.61	0.00	46.0	Variable
Total per cent[5]	100.0	100.0	100.0	......	......	......	......	......	......	......	......
B. T. U. (calculated)	754	499	605	......	......	......	......	......	......	......	......
B. T. U. (observed)	......	......	......	683	322	641	680	1000	1390	153	240 (Average)
Retort temp.[2]	585	615	845								
Rate gas prod.[3]	0.534	0.267	0.267								
Rate oil prod.[4]	60.0	64.0	20.0	......							
Rate temp. rise (°F.)	3.00	2.67	7.32	2							
Per cent oil off	49.7	57.0	97.8								

---

[1] Analyses Nos. 1, 2 and 3 represent gas produced at different stages in the same retorting test.

[2] Determined as indicated in footnote [3], Table VI, page 23.

[3] In cubic feet per minute.

[4] In cubic centimeters per minute.

[5] H_2S not determined; may average as high as 8 per cent.

MAP OF NORTHWESTERN COLORADO

The shaded areas show the extent of the Green River formation in which the workable beds of oil shale occur.

RESULTS OF NINE OIL-SHALE RETORTING TESTS.

INTRODUCTION.

The studies of factors affecting the retorting of oil-shale undertaken by the United States Bureau of Mines and State of Colorado in April, 1920, have progressed to such an extent that the publication of results of preliminary tests seems justified. This paper, therefore, submits nine curves, and a summarized table, from which the curves were derived, showing relations between temperature, oil production, gas production, thermal value of gas, gas sampling, and in some cases, water production. It is pointed out that these tests were made with a horizontal rotary retort, and that the shales were dry distilled, no steam or other gas being used in the retort. The results presented herewith probably apply only for the retorting conditions adhered to in the respective tests.

EXPERIMENTAL PLAN.

The retorting tests at the Boulder Co-operative Oil-Shale Laboratory are planned to follow a definite program. A retorting test is made under a definite set of conditions and then the products are examined, so that the results of certain applied conditions may be known before the next test is begun. The purpose of the study is to determine those conditions most favorable for producing the highest yields of the best grade of oil from oil-shales. Therefore many variable conditions of retorting must be studied, and their effects on quantity and quality of products determined. Such a study will require a considerable period of time for completion, as the effects of the following variable factors must be determined:

A. Nature of the shale.
B. Rate of rise of retorting temperature.
C. Size of shale particles retorted.
D. Actual temperature range used in retorting.
E. Use of steam and other gases at various temperatures and pressures and in different amounts.
F. Use of pressures above or below atmospheric.
G. Material used in, and design of retorting equipment.
H. Means by which heat is applied to the shale.
I. Time retorting products are in contact with heated surfaces, or in other words, velocities of vapors through and from the retort.

In the studies under way each variable is changed according to a regular program until best results are obtained, then another variable is changed, the idea being that ultimately the work will enable definite conclusions to be drawn as to the proper combination of conditions necessary to produce best results.

DESCRIPTION OF EXPERIMENTAL WORK.

The material presented in this paper deals with the first nine retorting tests made in the Boulder laboratory, and may be considered a preliminary study. When the apparatus was first erected it was necessary to determine its flexibility, its behavior with shales of different richness, and its ability to operate under pressures different from atmospheric, before a definite program of work could be undertaken. Therefore, in the first four distillations shown in Table VIII and in Curves 1 to 4, are results obtained while the retorting equipment was being tried out and the operators familiarizing themselves with the apparatus. Tests Nos. 5 to 8 inclusive represent the first four tests undertaken in the plan to determine the effects of various rates of heating, all other conditions being kept as nearly constant as possible. Test No. 9 represents the results on a lean shale which was examined to secure information not particularly in line with the plan of the program.

Since the data presented in this paper were obtained, many more retorting tests have been made, both in the Boulder Cooperative Laboratory and at the Intermountain Experiment Station of the Bureau of Mines, which is carrying on similar investigations, as has been noted. The study is by no means complete, and cannot be expected to be complete for considerable time. Good progress has been made, however, and in a short time it will be possible to present a paper giving results of many more retorting tests with complete examinations of the products made in each. The present paper will indicate the trend of the work and draw some interesting and valuable conclusions.

It will be noted that the shale used in the first four tests yielded on assay 42.7 gallons of oil to the ton, but as it was not possible to secure a large supply of this grade of shale at a reasonable cost, it was decided to carry out the investigation with the shale used in tests Nos. 5 to 8 inclusive. A large quantity of this shale has been secured and it is now being used. It will also be used in future work.

Retort temperatures given in the table and figures were determined by means of a thermo-couple placed in the center of the retort along the horizontal axis. It is known that temperatures along the line of the horizontal axis of the retort are practically uniform, but it is likely that temperatures so determined are considerably less than the actual temperatures of the shale distilling in the retort. The retort is now being equipped so that the actual shale temperature may be determined.

In all cases 75 pounds of shale were charged into the retort, making a layer 3½ inches thick at its greatest depth in the retort. The shale was crushed to pass a 2-inch opening, and all particles smaller than ¼ inch were screened out.

TABLE VIII.

SUMMARY OF NINE RETORTING TESTS ON COLORADO OIL-SHALE.[1]

Test Number	1	2	3[2]	4[3]	5	6	7	8	9
Shale charged, pounds	75.0	75.0	75.0	75.0	75.0	75.0	75.0	75.0	75.0
Real average rate of heating, deg. F. per minute[4]	3.88	6.12	6.82	8.16	3.56	3.49	3.46	3.36	6.38
Apparent rate of heating, deg. F. per minute[4]	2.51	6.12	4.36	6.58	3.42	3.49	3.46	3.08	6.38
First gas appeared at, deg. F[5]	320	......	......	......	210	235	235	235	266
First water appeared at, deg. F.[6]	......	......	......	350	......	310	325	350	300
First oil appeared at, deg. F[7]	363	375	350	350	460	465	455	455	441
Last gas appeared at, deg. F		Gas production would continue beyond any temperature reached.							
Last water appeareed at, deg. F.[8]	......	......	......	......	......	540	495	490	480
Last oil appeared at, deg. F	1030	1040	870	800	1180	1100	1130	1110	1090
Final temperature of retort, deg. F	......	......	1005	1050	1290	1180	1200	1180	1180
Range of oil production, deg. F	667	665	520	475	720	635	675	655	649
Total water yield, cubic centimeters[9]	431	486	371	266	544	825	1068	521	550
Total oil yield, cubic centimeters[10]	4789	5394	4124	3575	3596	4109	3798	3914	2472
Total gas yield, cubic feet	36.0	82.5	68.8[11]	119.5	132.8	36.3	81.5	93.5	....
Oil yield on assay, gals. per ton	42.7	42.7	42.7	42.7	37.0	37.0	37.0	37.0	28.0
Oil yield this test, gals. per ton	33.7	38.0	29.1	25.2	25.4	28.9	26.7	27.4	17.4
Per cent oil recovery[12]	78.0	89.1	68.2	59.1	68.5	78.2	72.2	74.5	61.5
Gas yield, cu. ft. per ton	958.5	2205.0	1838	3185	3550	967	2177	2496	....

[1] In horizontal rotary retort.

[2] Vacuum during test, 18.5 inches of mercury.

[3] Vacuum during test, 5.5 inches of mercury.

[4] See explanation, page 39.

[5] Approximate; these are temperatures in center of retort when first gas was noticeable from condensers.

[6] Temperature in center of retort when first water collects from condenser.

[7] Temperature in center of retort when first oil collects from condenser. Small scale tests on these shales indicate that the first oil is yielded when the actual shale temperature has reached about 350° C.

[8] Free water from condenser; water is produced all during the run but the greater part of it is in emulsion with oil.

[9] Includes total water suspended in oil and some condensed water from cleaning out with steam.

[10] Corrected for water in suspension (see explanation, page 41).

[11] Gas meter broke during run (see Fig. 3).

[12] Based on recovery in assay retort as 100 per cent.

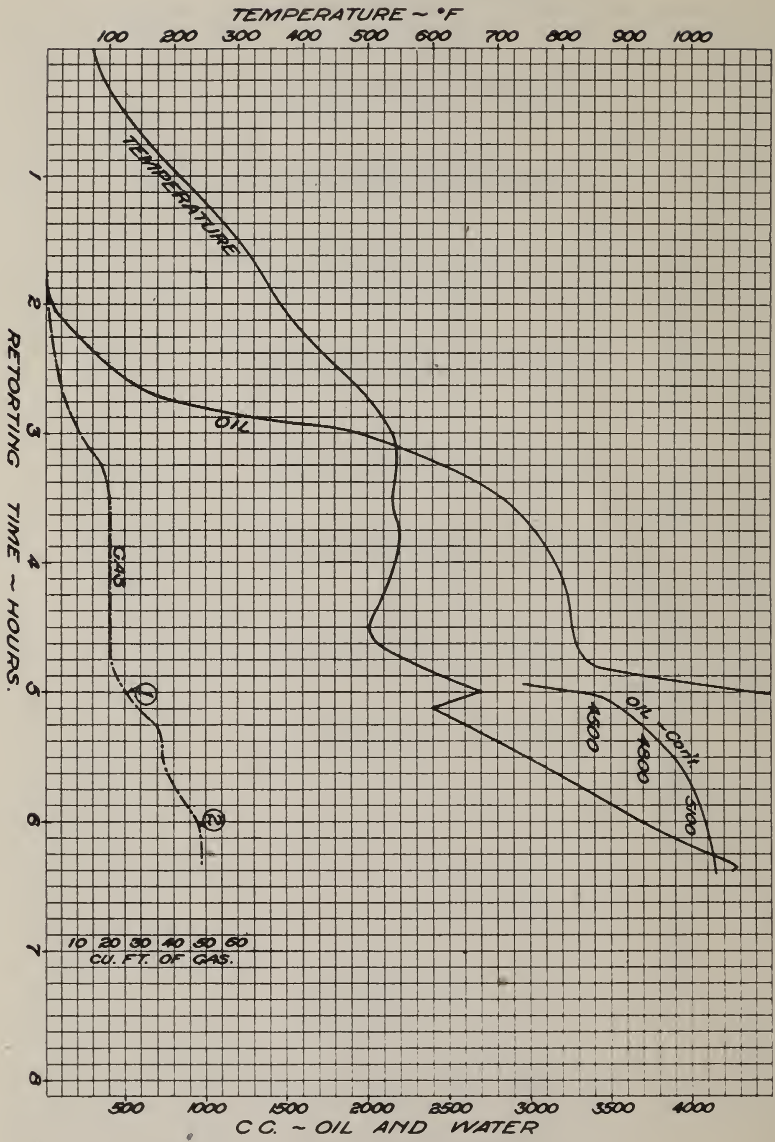

Figure 1.—Graphic Representation of Retorting Test No. 1. Date: May 20, 1920. Colorado Oil Shale. Assay, 42.7 gallons oil per ton. Average heating rate, 3.88° F. per minute. Pressure, atmospheric. Oil curve not corrected for water in suspension; see Table VIII and page 41. Total water in suspension this run, 431 cc., or 8.26 per cent.

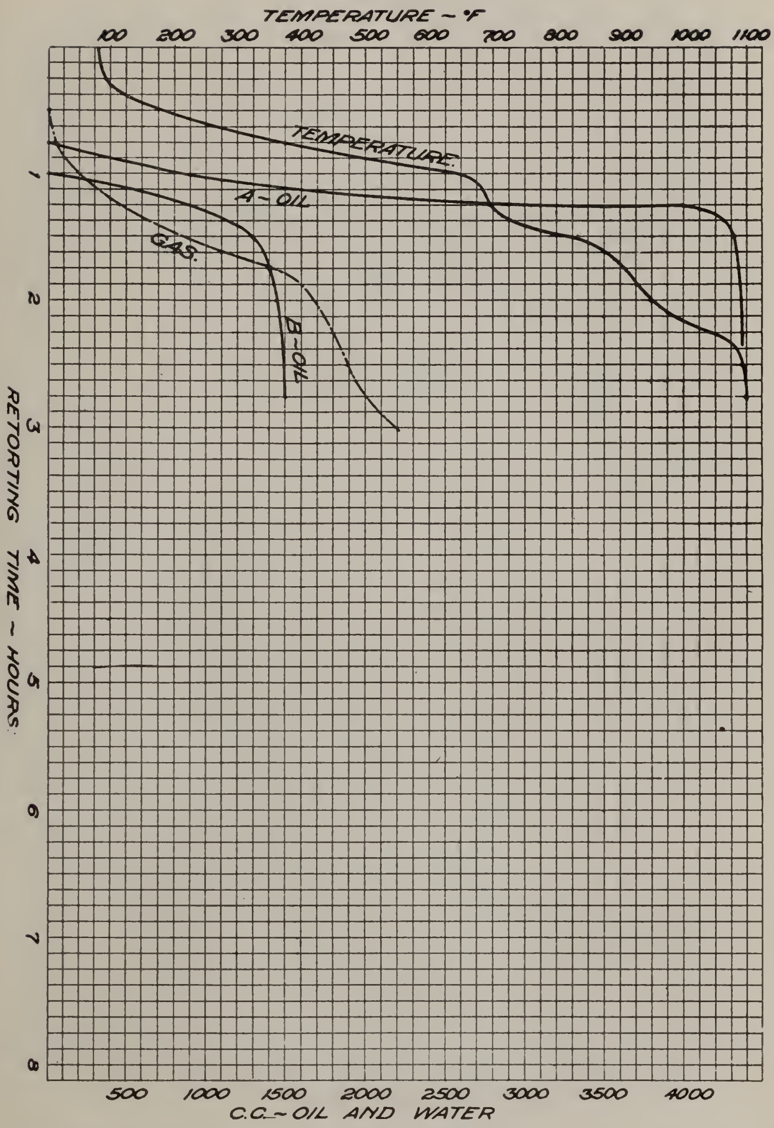

Figure 2.—Representation of Retorting Test No. 2. Date: May 25, 1920. Colorado Oil Shale. Assay, 42.7 gallons oil per ton. Average heating rate, 6.12° F. per minute. Pressure, atmospheric. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water in suspension. See Table VIII and page 41. Total water in suspension this run, 486 cc., or 8.26 per cent.

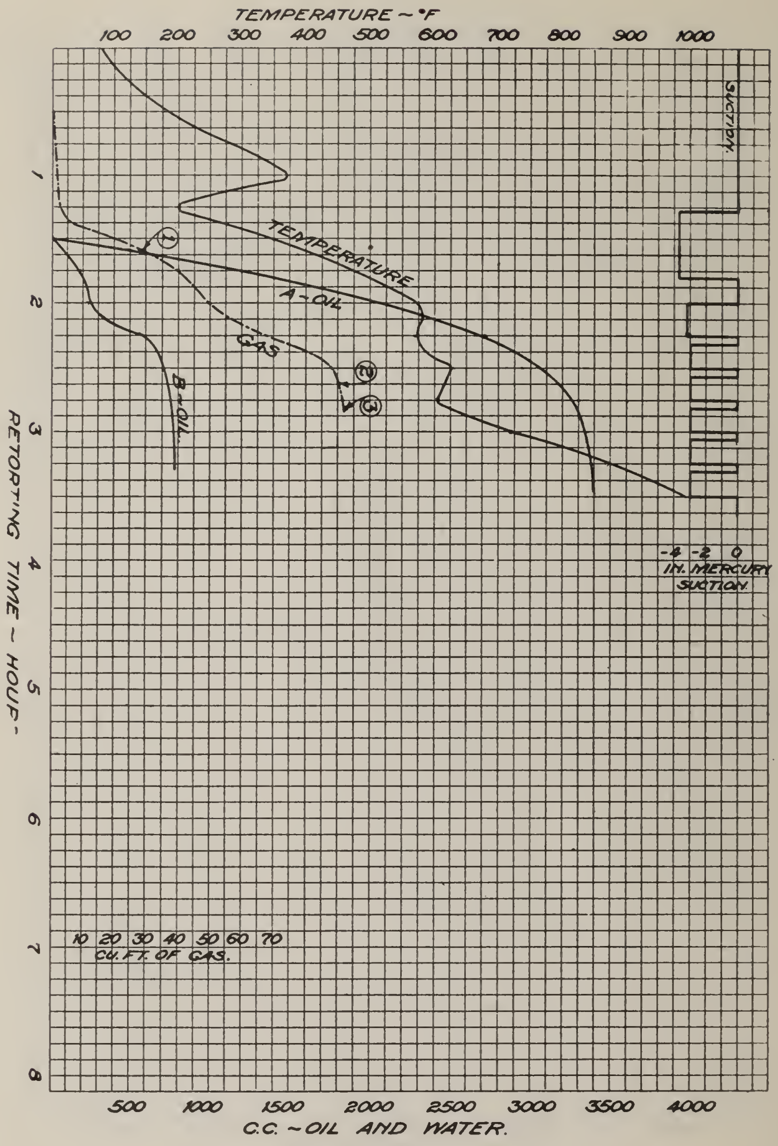

Figure 3.—Graphic Representation of Retorting Test No. 3. Date: May 29, 1920. Colorado Oil Shale. Assay, 42.7 gallons oil per ton. Average heating rate, 6.82° F. per minute. Pressure, reduced, as indicated. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water in suspension. See Table VIII and page 41. Total water in suspension this run, 371 cc., or 8.26 per cent. (1) Calorific value of gas, 482 B. t. u.; (2) Calorific value of gas, 492 B. t. u.; (3) Meter broke.

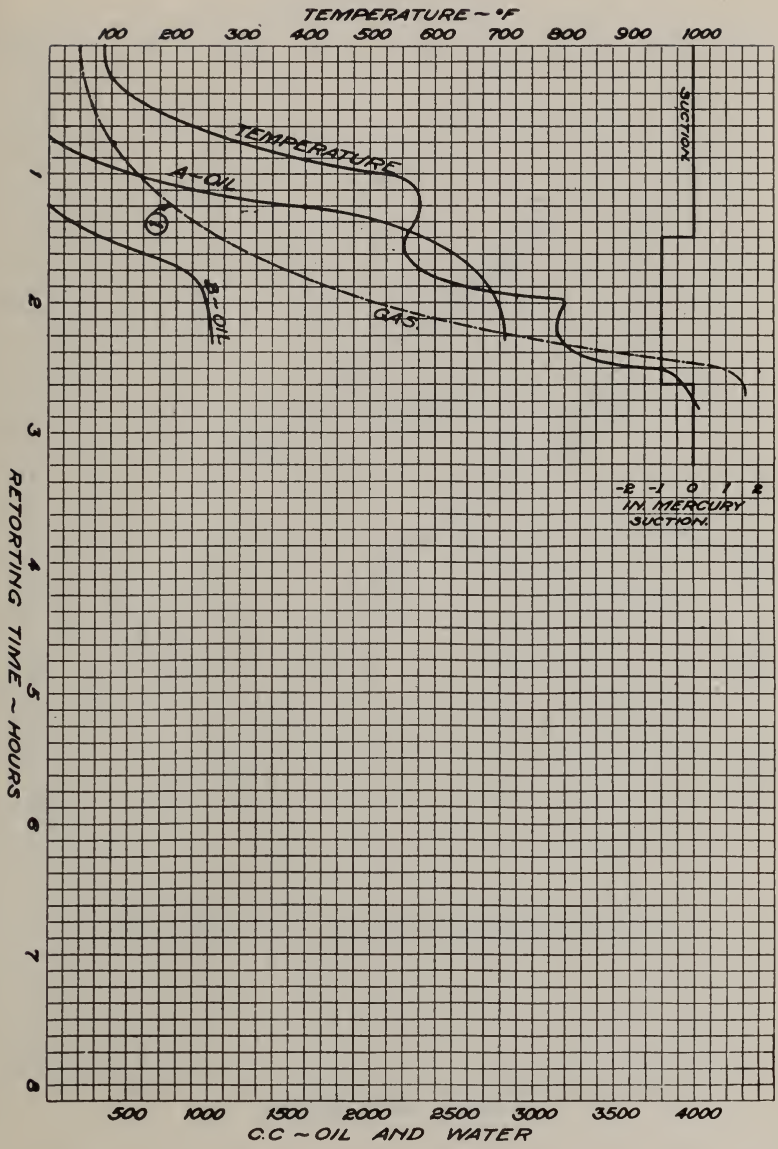

Figure 4.—Graphic Representation of Retorting Test No. 4. Date: August 20, 1920. Colorado Oil Shale. Assay, 42.7 gallons oil per ton. Average heating rate, 8.16° F. per minute. Pressure, reduced as indicated. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water in suspension. See Table VIII and page 41. Total water in suspension this run, 266 cc., or 6.93 per cent. (1) Calorific value of gas, 1045 B. t. u.

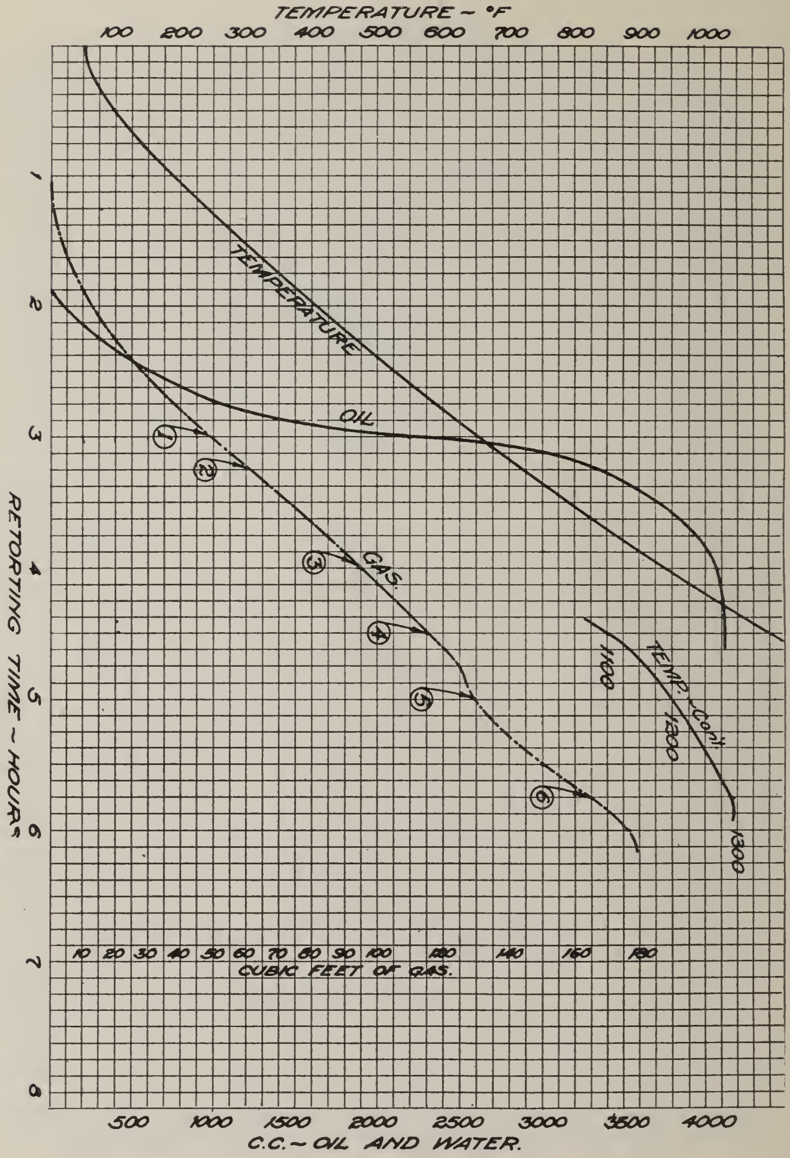

Figure 5.—Graphic Representation of Retorting Test No. 5. Date: September 30, 1920. Colorado Oil Shale. Assay, 37.0 gallons oil per ton. Average heating rate, 3.56° F. per minute. Pressure, atmospheric. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water and suspension. See Table VIII and page 41. Total water in suspension this run, 573 cc., or 13.9 per cent. (1) Calorific value of gas, 274 B. t. u. (2) Gas sample taken. (3) Gas sample taken. (4) Gas sample taken. (5) Calorific value of gas, 141 B. t. u. (6) Gas sample taken.

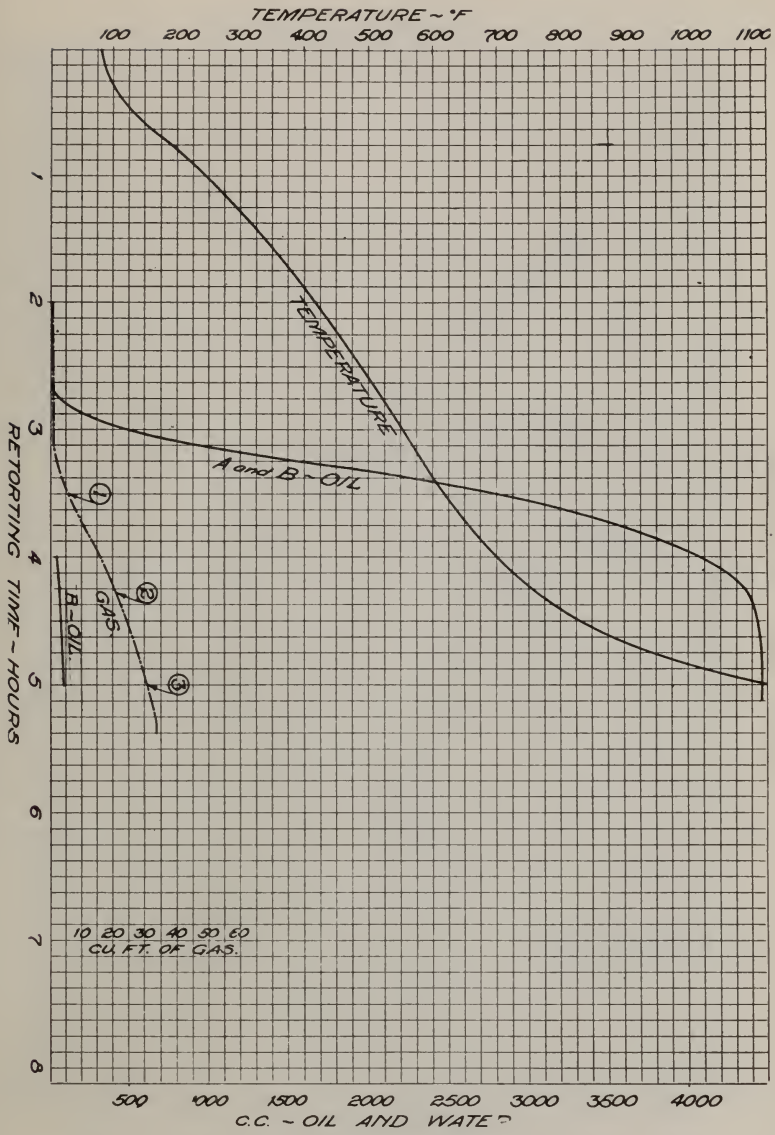

Figure 6.—Graphic Representation of Retorting Test No. 6. Date: October 9, 1920. Colorado Oil Shale. Assay, 37.0 gallons oil per ton. Average heating rate, 3.49° F. per minute. Pressure atmospheric. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water in suspension. See Table VIII, and page 41. Total water in suspension this run, 413 cc., or 9.18 per cent. (1) Heating value of gas, 1,049 B. t. u. Gas sample taken. (2) Heating value of gas, 843 B. t. u. Gas sample taken. (3) Heating value of gas, 285 B. t. u. Gas sample taken.

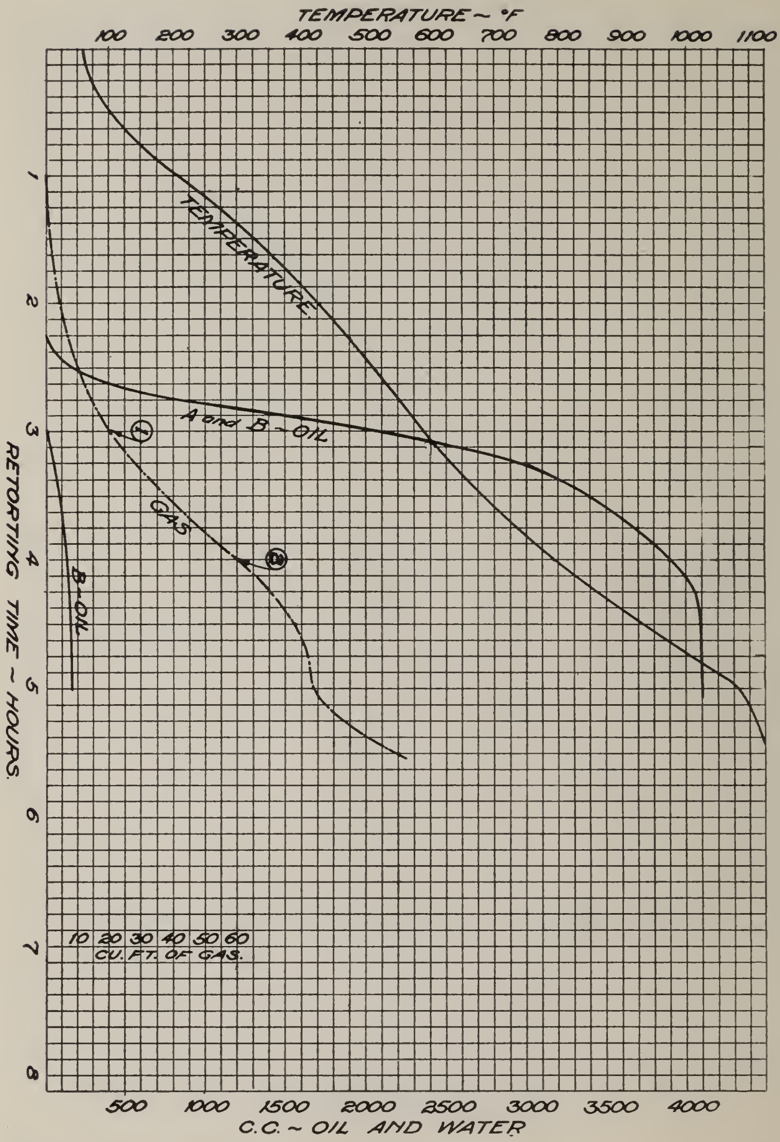

Figure 7.—Graphic Representation of Retorting Test No. 7. Date: October 13, 1920. Colorado Oil Shale. Assay: 37.0 gallons oil per ton. Average heating rate, 3.46° F. per minute. Pressure, atmospheric. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water in suspension. See Table VIII and page 41. Total water in suspension this run, 418 cc., or 9.31 per cent. (1) Heating value of gas, 708 B. t. u. (2) Heating value of gas, 817 B. t. u.

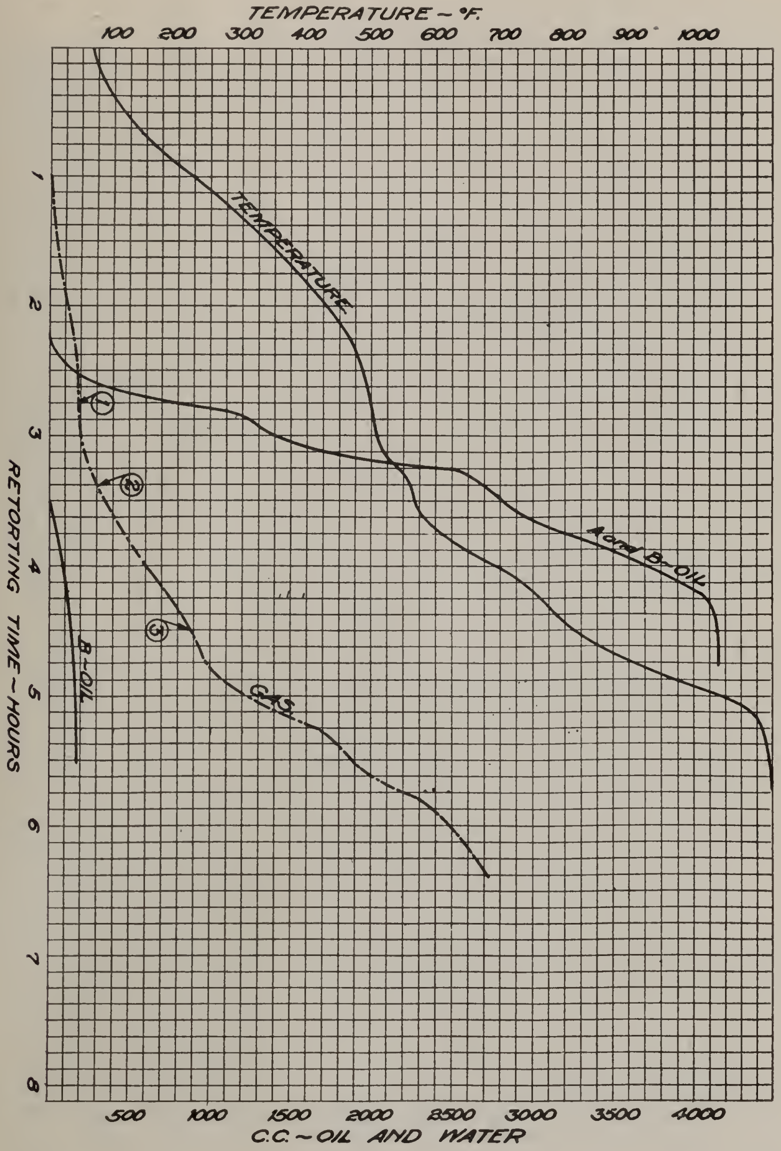

Figure 8.—Graphic Representation of Retorting Test No. 8. Date: October 15, 1920. Colorado Oil Shale. Assay, 37.0 gallons oil per ton. Average heating rate, 3.36° F. per minute. Pressure, atmospheric. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water in suspension. See Table VIII, and page 41. Total water in suspension this run, 318 cc., or 7.56 per cent. (1) Heating value of gas, 994 B. t. u. (2) Heating value of gas, 1,126 B. t. u. (3) Heating value of gas, 741 B. t. u.

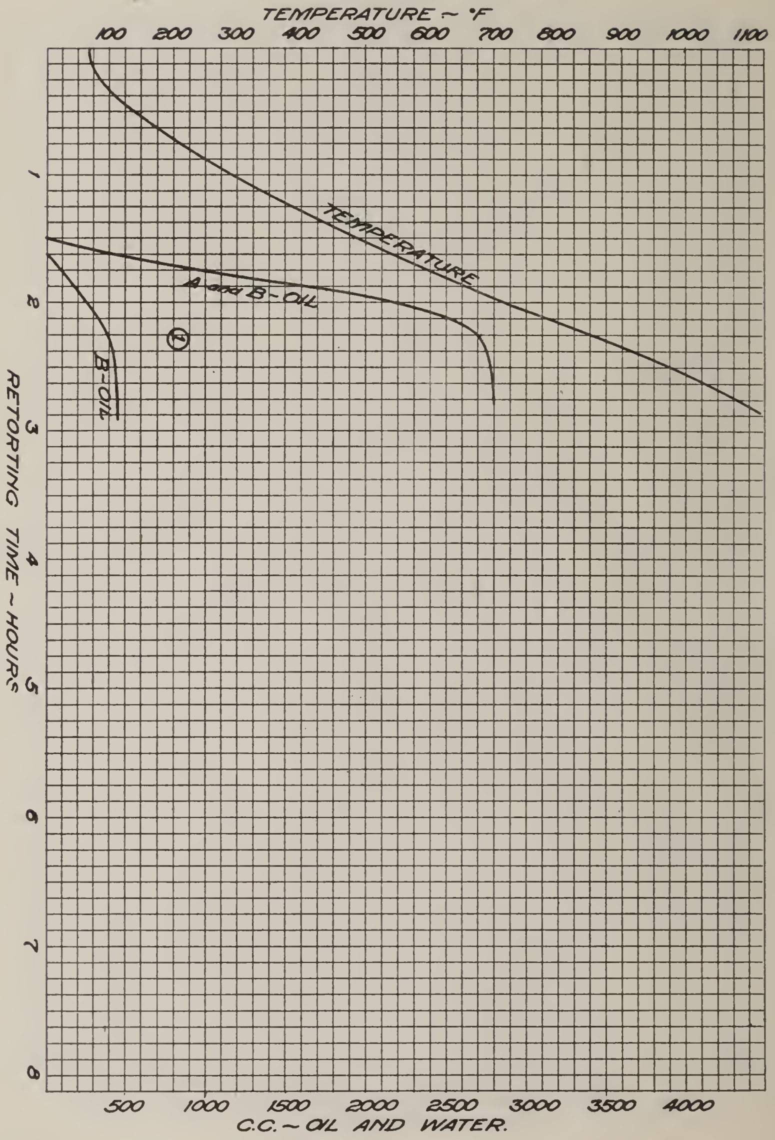

Figure 9.—Graphic Representation of Retorting Test No. 9. Date: October 27, 1920. Colorado Oil Shale. Assay, 28.0 gallons oil per ton. Average heating rate, 6.38° F. per minute. Pressure, atmospheric. A Oil is oil condensed in air cooled condenser. B Oil is oil condensed in water cooled condenser. Oil curves not corrected for water in suspension. See Table VIII, and page 41. Total water in suspension this run, 313 cc., or 11.25 per cent. (1) Heating value of gas, 312 B. t. u.

DISCUSSION OF RETORTING TESTS.

As has been mentioned, the first four tests were not run according to any definite program, and so many variables entered into these tests that it is impossible to draw conclusions as to the effect of any one variable. In tests Nos. 5 to 8 all conditions were held as nearly constant as possible.

The first four tests were made with the retort rotating at seven revolutions per minute. This rate was found to be too rapid, as it produced a decided ball-mill effect on the shale. Consequently the rate of rotation has been reduced to 3.75 revolutions per minute, which seems to be quite satisfactory, and was adhered to in the other tests reported. This rate will be used in the future.

The first two tests were made under atmospheric pressure, and it was noticed that a certain amount of dust was carried over into the condensing system and pump. In tests Nos. 3 and 4, made under vacuum, carrying over of the dust became so serious that a drum-head, made of iron screen and packed with steel wool, was placed in the discharge end of the retort, just in front of the vapor outlet. This seems to prevent trouble due to dust, but as the screen-head apparently has a bad effect on the oil, evidently producing cracking, it may be necessary to remove it. With the slow rate of rotation now being used it is believed that the use of the screen-head will be unnecessary, unless the retort is operated under reduced pressure.

Tests Nos. 3 and 4 were made under vacuum, as may be noted in Table VIII. Rather unexpected results were obtained. It was believed that a higher recovery of oil and a smaller amount of gas would be obtained from the shale under reduced pressure. Entirely contrary results were obtained. Since only two vacuum tests were made it is difficult to account for this fact, and no attempt is made to explain it, except that excessive cracking or incomplete condensation may have been responsible for the unexpected results. At the proper time in the course of the experiments the effects of reduced pressures will be carefully studied, making it possible to draw conclusions. The vacuum tests made indicate that the retort can be operated successfully under greatly reduced pressure without leakage. Tests Nos. 3 and 4 indicate also that the shale begins to produce oil at a lower temperature under reduced pressure than under atmospheric pressure.

It will be noted in Table VIII that the rate of heating is given as real average rate of heating and also in terms representing apparent average rate of heating. The first takes no account of the time when temperatures remained constant or fell, but considers only that period during which the temperature was actually rising and the total gross increase of temperature during that time. The second is obtained by dividing the total net temperature rise by the number of minutes required during the test to reach the maximum temperature. It will be noted that the figures representing these two calculated rates are very nearly the same in the later tests, but the earlier ones show considerable variation, due to inexperience with the apparatus.

CONCLUSIONS.

Examination of Table VIII and the curves indicates the following:

A. *Initial temperatures*[1].

The evolution of *gas* in noticeable quantity commences between 200° and 250° F., and usually close to 235° F. (113° C.).

Water probably starts distilling at about the same temperature as the gas and appears as condensed water from the condenser when the retort temperature is about 325° F. (163° C.).

Oil begins to flow from the condenser when the temperature in the center of the retort reaches about 450° F. (232° C.). It is noteworthy that the shale used in tests Nos. 1 to 4 began to produce oil at a lower temperature than the other shales used. Vacuum reduced the initial oil production temperature considerably (see tests Nos. 3 and 4).

B. *Range of production.*

Gas production will continue considerably above the highest temperature used in these tests. The volume will probably increase for some time, and its heating value decrease. (See paper on shale gas, page 24.)

Water is produced throughout the oil production range, but above 500° F. (260° C.) the water is usually emulsified with the oil, and cannot be separated by gravity settling at ordinary temperatures.

The average range for oil production is about 625° F. (350° C.), all of it being distilled off at a temperature between 1100° and 1150° F. (593°-621° C.). Nearly all the oil (about 96 per cent) is produced before the temperature reaches 900° F. (482° C.).

C. *Yields.*

The total *gas yield* in the different tests varies from about 1,000 cubic feet per ton of shale to nearly 3,600 cubic feet, and averaged 1,770 cubic feet per ton. In no case was gas production forced to the limit.

The *oil yields* show a considerable variation ranging from 59 to 89 per cent, based on assays. No gas scrubbers were used in tests Nos. 1 to 4.

Water yields vary considerably, as different parts of the shale samples contain different percentages of water. In some of the tests reported water yields are higher than actual yields from the shale, as it is often necessary to clean the apparatus with steam at the end of the run to remove all the oil. This sometimes introduces an unmeasured amount of water into the results, especially if the steam and condensed oil emulsify.

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[1] See footnotes, Table VIII, on temperature measurement. Particular emphasis is directed to the position of the pyrometer.

Much water is produced in emulsion with the oil. Oil yields shown in Table VIII are corrected for water in emulsion, but yield curves in Figures 1 to 9 are not so corrected, as it was not possible to determine the percentage of water in emulsion in the oil as the oil accumulated in the measuring receiver. Percentage of emulsified and suspended water is, however, indicated on the curves.

D. *Rate of temperature rise.*

Under this heading only tests Nos. 1 and 2, and 5 to 8 inclusive, can be discussed, for reasons above presented. It will be noted that the rates of heating used do not vary widely, but generally it appears that a greater yield of oil is obtained with the slower rates of heating. The effect on the quality of the oil has not been clearly established, but it appears that oils of better quality are produced at slower rates of heating. It has been noted in every case that when very rapid rates of heating were employed, the oil had the odor of badly cracked oil.

The effect on gas production is apparently as is to be expected, that is, smaller quantities of gas are produced at the lower rates of heating.

FUTURE RETORTING WORK.

The retorting work now under way is to determine the effects of several variables on the products of retorting oil-shales. The effect of different rates of heating are being studied first, and tests are now being made with rates much more varied than reported in this paper. It has been suggested that a uniform rate of heating may not be a desirable function on which to base work of this sort, and that a uniform rate of oil production would be better. Theoretically this suggestion has much merit but when its commercial applications are examined it would seem to limit certain types of retorts by greatly complicating their structure, if indeed it does not absolutely bar them. Work will be continued using different uniform rates of heating on the same shale, and then it is planned to make a series of tests, using constant oil production as a basis.

After the most favorable heating rate has been determined for the particular shale under examination, other factors will be varied, as mentioned on page 27. It is particularly desired to determine the effects of different sizes of shale particles on the products made at different heating rates. Any suggestions and data bearing on this work from persons interested in oil-shale will be appreciated.

ANALYTICAL DISTILLATION OF SHALE OIL FROM COLORADO OIL-SHALE.

INTRODUCTION.

After each retorting test at the Boulder Co-operative Oil-Shale Laboratory, samples of the various products collected during the run are examined in the laboratory. An analysis is made of the gas evolved during retorting, and its calorific value is determined. Condensed water from the retort condensers is examined primarily to determine content of nitrogen. Liquids from the oil and water scrubbers are tested for content of gasoline and ammonia, respectively. Spent shale from the retort is assayed to determine completeness of retorting, and a proximate analysis is made on it. Finally the oil recovered during the run is fractionally distilled, first at atmospheric pressure, and then under reduced pressure, and the fractions are examined.

In some cases one overall sample of the oil produced during the run is taken. In others several samples are taken during the course of the distillation of the shale. Proper examination of the several samples indicates whether the oil produced from the shale changes during the retorting period.

This paper presents a series of analyses of oils, all produced from the same shale during the same retorting test. The large horizontal rotary retort was charged with Colorado oil-shale, obtained near DeBeque, and retorting carried out in the usual manner. The oil production was allowed to accumulate until the retort temperature, read as previously indicated, had reached 269° C. (516° F.), then the oil receiver was changed. Likewise receivers were changed when the temperature had reached 285° C. (545° F.), 322° C. (612° F.), and when the run was completed. Thus four oil samples were obtained, the first representing oil production from start of distillation to 269° C., the second oil produced while the retort temperature increased from 269° to 285° C., and so on.

This report deals with only one such test, but several others have been made, oil samples being taken at different temperatures and the retort being operated under different conditions. All these data are being compared and correlated. Analyses of oils made under different retorting conditions are valuable as they not only permit a comparison of products made under various conditions, but show the effect of any single condition on the quality of the product.

In the near future a complete report will be issued giving details as to the analyses of the various oils produced during the first series of ten runs in the Boulder retort. This report will also attempt to indicate the specific effect of different retorting conditions on the quality of the oils produced.

LABORATORY PROCEDURE FOR EXAMINING SHALE OILS.

The method used in examining shale oils is practically that used by the United States Bureau of Mines in examining petroleum, and consists essentially of the following steps:[1]

1. The sample as received is heated in the container until it is fluid. (Most shale oils are of the consistency of butter at temperatures ranging from 60° to 95° F.) The container is then shaken to thoroughly mix the contents and a small sample taken therefrom for determination of water percentage.

2. If the water determination indicates only a small percentage of water, the sample is allowed to stand in a tightly stoppered container in a warm place until the bulk of the water has settled out, then a 600 cubic centimeter sample is carefully drawn off the top and placed in a copper topping-still. The light oil and water are distilled from the sample, and the water separated from the light oil. After the remaining oil in the topping-still has cooled, the light oil is added to it, and thoroughly mixed. If the topping is carefully done, and cold water used in the condenser, the loss during topping-distillation will not amount to more than a few tenths of one per cent.

Many shale oils, however, contain a large percentage of water which will not separate on standing, and when such a sample is to be run, it is necessary to dehydrate the oil by means of a drying agent. In such cases the oil is placed in a strong steel container provided with a tightly fitting plug and thermometer-well, and five grams of calcium chloride are added for every cubic centimeter of water contained in the sample. The plug is then screwed down tightly and the container heated gradually until the contents have reached a temperature of 200° C. The container is now shaken until it has cooled to room temperature, when the contents may be removed. This method serves to dry thoroughly oils containing large amounts of water.

3. Specific gravity of the clean oil, dried by either of the above methods, is then taken at 15.56° C. (60° F.). Practically all shale oils are semi-solid at this temperature, and it is therefore necessary to use some type of specific gravity bottle adapted for use with solid oils and tars. The Barrett type of specific gravity bottle has been found satisfactory for this purpose.

4. The specific gravity of the oil having been determined, it is necessary to calculate the weight of oil corresponding to 300 cubic centimeters. This amount is weighed into the Bureau of Mines standard Hempel distilling flask. This flask, whose dimensions have been accurately fixed, consists of a spherical glass bulb of 500 cubic centimeter capacity, with a 10-inch vertical neck or column, and with a delivery tube springing from the neck nine

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[1] The apparatus and methods used in these tests with minor exceptions, have been developed in the Pittsburgh laboratory of the Bureau of Mines, and are fully described in Bulletin 209 of the Bureau of Mines, "The Analytical Distillation of Petroleum" by E. W. Dean, soon to be issued.

inches up the column at an angle of 15 degrees from horizontal. After the oil has been poured into the flask (it is often necessary to heat the oil to a point of fluidity before it can be poured into the flask) an 8-inch column of iron ''jack chain'' to serve as a fractionating column is placed in the vertical neck. A cork, through which a thermometer passes, is fitted into the top of the vertical neck of the flask. The thermometer is so placed that the top of the mercury bulb is on a level with the bottom of the opening of the delivery tube. The delivery tube is connected to the condenser with a well-fitting cork. The condenser consists of a vertical three-bulb staggered glass tube, of standardized dimensions, set in an insulated jacket, which at the start of the distillation is filled with water and shaved ice.

5. In setting up the distilling outfit all joints are luted with a paste of litharge and glycerine. The Bureau of Mines usually employs an electric resistance heater, controlled by a variable resistance, to heat the flask. Distillation is allowed to proceed at the rate of about two cubic centimeters a minute, fractions being separated at every even 25° C. interval. The temperature of the first drop over is noted. The distillation is continued until the vapor temperature reaches 275° C., all fractions taken during the intervals of 25 degrees temperature rise being kept separate in stoppered glass tubes.

6. The flask is next allowed to cool, the column of chain is removed and two cones of copper gauze are inserted in its place. These cones are placed about one inch apart in the middle of the neck. The thermometer is then replaced, the flask connected to the condenser as before, and the delivery end of the condenser connected to a vacuum receiver. Fractions may now be obtained under vacuum without breaking the vacuum to change receivers. To prevent paraffin wax solidifying in the tube, the water in the condenser jacket is slowly heated with an electric immersion heater until it is nearly at the boiling point when distillation is complete. Vacuum distillation is conducted at a pressure of 40 millimeters of mercury, and at the same rate as during the air distillation. Cuts are made at a vapor temperature of 200° C., and at every 25 degrees up to 300° C., when the distillation is stopped. The residuum in the flask is allowed to cool, and its specific gravity and setting point are determined.

7. Fractions taken at atmospheric pressure are examined separately; volume and specific gravity of each fraction at 15.56° C. being determined.

8. Either the percentage of unsaturation of each fraction is determined, or the percentage of unsaturation of the combined fractions distilling up to 200° C. is determined. The separate or combined fractions distilling from 200° C. up to 275 C°. are similarly tested. Percentage of unsaturation is the percentage soluble in sulphuric acid of 98 per cent strength. Briefly, the method of determining unsaturation consists of carefully mixing 5 cubic centimeters of the oil with 10 cubic centimeters of the

acid, in a small bottle with graduated neck, keeping the bottle well cooled while mixing the contents. The bottle is then placed in a centrifuge and centrifuged until a complete separation of the oil not acted on by the acid has been effected.[1]

While it is realized that this method does not accurately determine the absolute percentage of unsaturated hydrocarbons in the shale oil fractions, nevertheless close checks and corresponding results can be obtained. The method is quite valuable for purposes of comparison because it indicates at least the comparative order of refining loss that the oil will suffer. Under the conditions of the test, sulphuric acid probably does not remove all the unsaturated hydrocarbons of the olefin series, and probably does remove some of the higher members of the saturated hydrocarbon series. The acid also removes nitrogen compounds from the oil. The acid, however, does remove all the hydrocarbons that are most objectionable in refined products, although commercial refining losses can be expected to be considerably lower than the unsaturated percentages indicated in the tables.

9. The fractions taken under reduced pressure are also examined separately. Volume and specific gravity at 15.56° C. are determined, using the specific gravity bottle for those fractions that are solid at that temperature. A Westphal specific gravity balance can be used for all fractions that are entirely fluid at the above temperature.

10. Setting points of the vacuum fractions are determined by freezing a drop of the oil on the extreme end of the bulb of a cooled thermometer, *inverting* the thermometer and rotating it about a vertical axis while the temperature is allowed to rise at the rate of 1° C. per minute. In most cases the drop of oil will melt sharply at a definite temperature and flow down the thermometer bulb. This temperature is the setting point, and gives an indication of the paraffin wax content of the fraction. In the case of some residuums, particularly if they contain much asphalt, the setting point cannot be determined accurately by this method.

11. The viscosities of the crude oil, and of the fractions taken off under vacuum, are taken at 60° C. (140° F.) by a Saybolt Universal Viscosimeter, or a glass pipette viscosimeter giving results that can be converted into Saybolt readings.

INTERPRETATION OF RESULTS OF DISTILLATION ANALYSES.

The following is quoted from a paper by Dean[2], indicating how the results of distillations are interpreted when petroleum oils are examined.

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[1] See Dean, E. W., and Hill, H. H., The determination of unsaturated hydrocarbons in gasoline, Bureau of Mines Tech. Paper 181, 1917, for details of this method.

[2] Dean, E. W., Properties of typical crude oils from the eastern producing fields of the United States, Bureau of Mines, Reports of Investigations, Serial No. 2202, January, 1921, 3 p.

"The methods employed by the Bureau of Mines for the distillation analysis of crude petroleum have not been developed with the idea of obtaining figures that parallel the results of actual refinery practice. As refinery practice has never been standardized, it has been necessary to select a fundamentally reproducible basis of comparison, rather than attempt to work in terms of yields and properties of commercial products.

"The chief value of the present report lies in the fact that it permits a reasonably adequate comparison of different crude oils on the basis of fundamental, physical and chemical properties.

"It is believed while the most satisfactory use of the figures involves a comparison, there is also a need for some sort of 'rough and ready' interpretation in terms of commercial products. Therefore, the author has employed the following classification which, even if it has no other justification, is convenient because it permits discussion in terms of 'given names.'

1. "The sum of all fractions distilling at atmospheric pressure below 200° C. (392°F.) is reported as *gasoline and naphtha.*

2. "The sum of all fractions distilling at atmospheric pressure between 200° C. (392° F.) and 275° C. (527° F.) is reported as *kerosene.*

3. "The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) of less than 50 seconds is reported as *gas oil.*

4. "The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) between the inclusive limits of 50 and 99 seconds is reported as *light lubricating distillates.*

5. "The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) between 100 and 199 seconds inclusive is reported as *medium lubricating distillate.*

6. "The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) of 200 seconds or more, is reported as *viscous lubricating distillate.*"

In the case of shale oils, the above interpretation must be applied with considerable care. In the first place, the unsaturation percentages of the crude naphtha and kerosene fractions are usually very high, indicating a high refining loss, and therefore the percentages of finished gasolines and kerosenes will be considerably less than those indicated by the tables. How much the refining loss will be is as yet unknown.

In the case of shale oil fractions taken off under vacuum, the term *gas oil* can probably be applied as above, but since little is known as yet of the properties of lubricating oils made from shale oil, a distillation analysis will not be of much value at the present time. Later on, of course, when shale oils are actually refined and used, results of the examination of lubricating fractions of shale oils produced by laboratory methods can be compared with the oils in use, and a reference point established. At present all fractions distilling under vacuum above 225° C. are classified as crude lubricating distillates, no attempt being made to classify further. About the only conclusion which can be reached at present is that from the standpoint of viscosity; some lubricating fractions seem suitable for making satisfactory lubricating oils. The possible durability of such lubricating fractions in actual use is yet to be determined, and durability, after all, is the property that deserves major emphasis in considering a lubricating oil.

The setting points of the higher boiling vacuum fractions indicate that a good percentage of paraffin wax may be obtained from the shale oils examined. Similar setting points for fractions of the Scotch shale oils, shown on page 50, are of interest in this connection.

On pages 48 to 50 inclusive are given Tables IX-A to IX-F showing the results of analytical distillations of the oils referred to.

TABLE IX-A.

ANALYTICAL DISTILLATION OF SHALE OIL FROM COLORADO OIL-SHALE.

Sample No. B-003 (81).
Oil-shale from DeBeque, Colorado. First fraction off retort.
Distilled in horizontal rotary retort. Retort temperature up to 269° C.
Specific gravity oil, 0.937. Baume gravity, 19.4.
Water in oil, 19.56 per cent. Setting point [1] Viscosity [1]

Distillation, Bureau of Mines Hempel Method.

Air distillation: barometer, 645 mm. First drop, 52° C. (126° F.).

Temperature, deg. C.	Per cent cut.	Sum per cent.	Sp. gr. cut.	Deg. Be. cut.	Unsaturation per cent.	Viscosity at 60° C.	Setting point, deg. C.	Temperature, deg. F.
Up to 50	....	....	[1]	[1]	[1]			Up to 122
50– 75	0.34	0.34						122–167
75–100	.27	.61						167–212
100–125	.95	1.56						212–257
125–150	3.22	4.78						257–302
150–175	3.87	8.65	0.817	41.4	50.6	......	......	302–347
175–200	4.83	13.48	.839	36.9	52.8	......	......	347–392
200–225	6.60	20.08	.858	33.2	[1]	......	......	392–437
225–250	7.48	27.56	.875	30.0	55.8	......	......	437–482
250–275	10.54	38.10	.890	27.3	57.8	......	......	482–527
Vacuum distilation at 40 mm.								
Up to 200	2.55	2.55	.908	24.2	......	44	[1]	Up to 392
200–225	7.75	10.30						392–437
225–250	8.26	18.56	.931	20.4	......	61	17.5	437–482
250–275	7.92	26.48	.941	18.8	......	106	27.0	482–527

Residuum: specific gravity 1.015; setting point 45° C.

[1] Not determined.

TABLE IX-B.

ANALYTICAL DISTILLATION OF SHALE OIL FROM COLORADO OIL-SHALE.

Sample No. B-002 (82).
Oil-shale from DeBeque, Colorado. Second fraction off retort.
Distilled in horizontal rotary retort. Retort temperature 269°–285° C.
Specific gravity oil, 0.984. Baume gravity, 12.3.
Water in oil, 4.75 per cent. Setting point, 10° C. Viscosity, [1].

Distillation, Bureau of Mines Hempel Method.

Air distillation: barometer 642 mm. First drop, 46° C. (114° F.)

Temperature, deg. C.	Per cent cut.	Sum per cent.	Sp. gr. cut.	Deg. Be. cut.	Unsaturation per cent.	Viscosity at 60° C.	Setting point. deg. C.	Temperature, deg. F.
Up to 50	tr.	......	0.768	52.3	46.0	......	......	Up to 122
50– 75	0.51	0.51						122–167
75–100	......	......						167–212
100–125	1.23	1.74						212–257
125–150	3.90	5.64						257–302
150–175	3.79	9.43	.803	44.4	49.6	......	......	302–347
175–200	5.07	14.50	.825	39.7	52.6	......	......	347–392
200–225	5.84	20.34	.843	36.1	[1]	......	......	392–437
225–250	7.79	28.13	.866	31.7	54.6	......	......	437–482
250–275	9.44	37.57	.884	28.4	59.0	......	......	482–527
Vacuum distillation at 40 mm.								
Up to 200	2.67	2.67	.906	24.5	......	43	......	Up to 392
200–225	7.90	10.57						392–437
225–250	8.26	18.83	.929	20.7	......	58	17	437–482
250–275	[1]	....	....	......	......	......	......	....

Residuum: specific gravity 1.173; setting point [1].

[1] Not determined.

TABLE IX-C.

ANALYTICAL DISTILLATION OF SHALE OIL FROM COLORADO OIL-SHALE.

Sample No. B-004 (83).

Oil-shale from DeBeque, Colorado. Third fraction from retort.
Distilled in horizontal rotary retort. Retort temperature 285°–322° C.
Specific gravity oil, 0.918. Baume gravity, 22.5.
Water in oil, 2.93 per cent. Setting point, 19° C. Viscosity at 60° C., 56.[1]

Distillation, Bureau of Mines Hempel Method.

Air distillation: barometer 640 mm. First drop 48° C. (118° F.)

Temperature, deg. C.	Per cent cut.	Sum per cent.	Sp. gr. cut.	Deg. Be. cut.	Unsaturation per cent.	Viscosity at 60° C.	Setting point. deg. C.	Temperature, deg. F.
Up to 50	tr.	tr.	0.772	51.4	49.0	......	......	Up to 122
50– 75	0.271	0.27						122–167
75–100	.542	.81						167–212
100–125	1.35	2.16						212–257
125–150	3.39	5.55						257–302
150–175	8.12	13.67	.823	40.1				302–347
175–200								347–392
200–225	4.57	18.24	.854	33.9	59.0	......	......	392–437
225–250	6.17	24.41	.872	30.6				437–482
250–275	6.85	31.26	.893	26.8				482–527
Vacuum distillation at 40 mm.								
Up to 200	2.58	2.58	.914	23.2	......	43	......	Up to 392
200–225	6.78	9.36						392–437
225-250	7.88	17.24	.923	21.7	......	59	17.0	437-482
250–275	8.27	25.51	.944	18.3	......	98	26.5	482–527

Residuum: specific gravity 1.004; setting point not determined, residuum very asphaltic.

[1] Viscosity at 100° F., 97.

TABLE IX-D.

ANALYTICAL DISTILLATION OF SHALE OIL FROM COLORADO OIL-SHALE.

Sample No. B-001 (84).

Oil-shale from DeBeque, Colorado. Fourth cut from retort.
Distilled in horizontal rotary retort. Retort temperature 599° C.
Specific gravity oil, 0.901. Baume gravity, 25.4.
Water in oil, 2.59 per cent. Setting point [1]. Viscosity at 60° C., 65.

Distillation, Bureau of Mines Hempel Method.

Air distillation: barometer 645 mm. First drop 41° C. (106° F.)

Temperature, deg. C.	Per cent cut.	Sum per cent.	Sp. gr. cut.	Deg. Be. cut.	Unsaturation per cent.	Viscosity at 60° C.	Setting point. deg. C.	Temperature, deg. F.
Up to 50	tr.	tr.	0.773	51.1	50.6	......	......	Up to 122
50– 75	0.33	0.33						122–167
75–100	.60	.93						167–212
100–125	2.13	3.06						212–257
125–150	2.77	5.83						257–302
150–175	4.00	9.83	.809	43.1	53.2	......	......	302–347
175–200	4.40	14.23	.835	37.7	53.8	......	......	347–392
200–225	4.60	18.83	.856	33.6	59.2	......	......	392–437
225–250	4.83	23.66	.879	29.3	64.4	......	......	437–482
250–275	6.10	29.76	.900	25.6	67.6	......	......	482–527
Vacuum distillation at 40 mm.								
Up to 200	2.21	2.21	.925	21.4	......	47	......	Up to 392
200–225	6.67	8.78						392–437
225–250	5.83	14.61	.941	18.8	......	65	17	437–482
250–275	8.72	23.33	.966	14.9	......	117	30	482–527

Residuum: Specific gravity [1]; setting point [1].

[1] Not determined.

TABLE IX-E.

ANALYTICAL DISTILLATION OF SHALE OIL FROM SCOTLAND[2].

Sample No. 0-011.
Oil-shale from Scotland. Water in oil, 0.13 per cent. Setting point 28° C.
Retorted in Pumpherston commercial retorts. Baume gravity, 29.6.
Specific gravity oil, 0.877. Viscosity at 60° C., 44.

Distillation, Bureau of Mines Hempel Method.

Air distillation: barometer 644 mm. First drop 49° C. (120° F.)

Temperature, deg. C.	Per cent cut.	Sum per cent.	Sp. gr. cut.	Deg. Be. cut.	Unsaturation per cent.	Viscosity at 60° C.	Setting point, deg. C.	Temperature, deg. F.
Up to 50	tr.	......	0.760	54.2	28.0	......	......	Up to 122
50– 75	0.13	0.13						122–167
75–100	.32	.45						167–212
100–125	.99	1.44						212–257
125–150	1.66	3.10						257–302
150–175	3.00	6.10	.785	48.3				302–347
175–200	6.12	12.22	.807	43.5				347–392
200–225	8.10	20.32	.826	39.5	34.0	......	......	392–437
225–250	6.65	26.97	.842	36.3				437–482
250–275	8.47	35.44	.857	33.4				482–527
Vacuum distillation at 40 mm.								
Up to 200	9.32	9.32	.872	30.6	......	38	......	Up to 392
200–225	5.27	14.59	.881	28.9	......	40	......	392–437
225–250	7.16	21.75	.892	27.0	......	46	24.5	437–482
250–275	6.13	27.88	.902	25.2	......	52	29	482–527
275–300	6.07	33.95	.911	23.7	......	60	34	527–572

Residuum: specific gravity 0.957 (16.3° Be.); setting point 41° C.

TABLE IX-F.

ANALYTICAL DISTILLATION OF CRUDE OIL FROM PENNSYLVANIA.

Sample No. 0-009.
Pennsylvania crude oil. Baume gravity, 42.4.
Specific gravity oil, 0.812. Viscosity at 60° C., 39.
Water in oil, trace.

Distillation, Bureau of Mines Hempel Method.

Air distillation: barometer 644 mm. First drop 26° C. (79° F.)

Temperature, deg. C.	Per cent cut.	Sum per cent.	Sp. gr. cut.	Deg. Be. cut.	Unsaturation per cent.	Viscosity at 60° C.	Setting point, deg. C.	Temperature, deg. F.
Up to 50	0.895	0.895	0.674	77.7	4.4	......	......	Up to 122
50– 75	1.612	2.507						122–167
75–100	4.08	6.59	.712	66.6				167–212
100–125	8.29	14.88	.733	61.0				212–257
125–150	5.46	20.34	.752	56.2				257–302
150–175	6.77	26.11	.763	53.5				302–347
175–200	5.82	31.93	.778	50.0				347–392
200–225	6.95	38.88	.789	47.4	3.6	......	......	392–437
225–250	6.42	45.30	.800	45.0				437–482
250–275	7.46	52.76	.812	42.4				482–527
Vacuum distillation at 40 mm.								
Up to 200	3.33	3.33	.826	39.5	......	39	......	Up to 392
200–225	7.75	11.08	.832	38.3	......	40	......	392–437
225–250	6.02	17.10	.841	36.5	......	45	15.5	437–482
250–275	5.37	22.47	.848	35.1	......	51	22.5	482–527
275–300	5.16	27.63	.859	33.0	......	67	30.0	527–572

Residuum: specific gravity 0.882 (28.7° Be.); setting point 18° C.

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[2] Does not include scrubber naphtha which amounts to 10% of crude.

COMPARISON OF ANALYSES OF SHALE OILS.

The prime purpose of this paper is to show by a presentation of experimental data that the nature of the oils produced from the same shales at different temperatures during the same run changes to a certain extent, but that the change is so small that it is of little commercial importance. While this conclusion can be drawn from the four analyses presented in this paper, it is emphasized that the conclusion has not been reached as a result of these four analyses alone. In fact some fifty samples of oil produced at various temperatures have been examined, and in no case has a striking difference been found in the oils produced at different stages during the same retorting test. However, striking differences have been observed between oils produced in different runs when some definite retorting condition, such as rate of rise of temperature, has been varied.

Tables IV-A to IX-F contain the records of analytical distillations of the four samples of oil referred to. In addition they give the distillation of a sample of commercial Scotch shale oil produced from Scotch shale in Scotland. Also there is presented the distillation analysis of a sample of high grade Pennsylvania crude oil. These last two analyses are inserted to show the difference between shale oil and petroleum, as indicated by distillations, and between shale oil produced in Scotland by commercial processes and the oil produced from Colorado oil-shale in the Boulder laboratory.

It is important to note that the operations at Boulder are of an experimental nature. Often, in experimental work, negative results are as valuable as positive, since negative results indicate what not to do, or show that the experiments are going in the wrong direction. The oils herein reported, therefore, may not be the best oils that can be made from the shale. As a matter of fact, better oils are constantly being produced as better conditions are being determined and applied. At the Intermountain Experiment Station of the Bureau of Mines, Salt Lake City, oils have already been produced by laboratory methods from Scotch shale that equal in every respect the shale oils produced in commercial operations in Scotland. So far, however, it has not been possible to produce equally satisfactory oils from American shales. A careful examination of American oil-shales by chemical and microscopic means has indicated that it will be a difficult matter to produce oils from American oil-shales that are of as good quality as Scotch shale oils, because of the differences in nature and origin of the different shales. Products from shales from different parts of the United States, and even from different parts of the Green River formation, show a marked difference although the shales were treated under identical conditions.

THERMAL CALCULATIONS ON THE RETORTING OF OIL-SHALES.

INTRODUCTION.

Engineers designing, or making calculations of capacities of oil-shale retorting equipment, will find it essential to determine the amount of heat necessary to retort a unit weight of a given shale. Also, it will often be desirable to know how much of that heat may be supplied by the shale gas and spent shale, either separately or in combination, or by fresh shale.

Using data already available, or presented in other parts of this bulletin[1], this paper presents calculations approximately indicating the following:

A. The theoretical amount of heat necessary to retort an oil-shale at various temperatures.

B. The total amount of heat necessary to retort an oil-shale at various furnace efficiencies.

C. The furnace efficiencies necessary if retorting is to be carried on with the shale gas, or shale gas and spent shale, without the introduction of other fuel.

METHOD OF MAKING CALCULATION OF HEAT REQUIRED FOR RETORTING.

In order to obtain a set of figures which might serve as a basis for estimating the value and suitability for retorting of shales of nearly any composition likely to be encountered, the following procedure was used:

The composition by weight (of residue, oil, water and gas) of four ideal shales calculated to represent probable average occurrences in the Colorado-Utah district was estimated. These ideal types of shales produced oil at rates ranging from 25 to 100 gallons of oil per ton, and probably cover nearly all workable oil-shales in this district. The composition of these ideal shales is given in Table X.

Having thus established the weights of each component of a set of shales having definite assays of oil, water and gas yield, the heat required for retorting was approximated as follows:

1. Certain *assumptions* were made.

   (*a*) A maximum temperature of retorting was assigned.

   (*b*) The specific heat of the shale was assumed not to vary up to 925° C.

   (*c*) A temperature was assigned as that of the beginning of the distillation.

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[1] Most of the factors necessary in making the calculations that follow have been given previously in publications of the authors or in this bulletin, and are summarized on page 62.

(d) The specific heat of the spent shale at comparatively high temperature is assumed to be the same as that determined at a lower temperature.

(e) The specific heat of the oil vapor is assumed to be very nearly that estimated for the oil at lower temperature.

(f) The latent heat of vaporization of the oil is assumed to be nearly that of its lighter constituents.

(g) The average specific heat of the permanent gases formed is assumed to be 0.35.

2. Certain *errors,* at present partly unavoidable, are neglected.

(a) Those errors involved in the assumption regarding specific heats, above.

(b) Errors involved in considering that no vapors of either water or oil, or gases, are given off below the temperature assigned as that of the beginning of distillation.

(c) Errors involved in considering that all vapors originate at the temperature of the beginning of distillation and are carried to the highest temperature of distillation before discharge from the retort.

(d) Errors involved in lack of quantitative information concerning the heat of decomposition or reaction of the shale kerogen, either positive or negative. The heat of reaction, however, is believed to be quite small.

Notwithstanding the assumptions made, and the errors known to be introduced, the results of the calculations agree well with experimental facts as is shown below.

3. The various factors entering into the heat calculation were assembled, and the following formula obtained for calculating the amount of heat required to retort a unit of oil-shale by dry distillation, at 100 per cent heating efficiency. The formula can be applied from the results of an assay on oil-shale in which oil and water yields and weight of spent shale have been determined, and weight of gas evolved arrived at by difference. Theoretical amount of heat, in small calories[1], required to retort oil-shale by dry distillation at 100 per cent efficiency =

$$454 \left\{ S \left[(T_1 - T_0) C' \right] + O \left[r' + (T_2 - T_1) C'' \right] + W \left[r'' + (T_2 - T_1) C''' \right] + G \left[(T_2 - T_1) C'''' \right] + R \left[(T_2 - T_1) C''''' \right] \right\}$$

in which:

454 = factor for converting pounds into grams. (If the weights used are expressed as grams this figure is eliminated.)

S = weight in pounds of shale considered.

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[1] If it is desired to convert this value into B.T.U.'s, divide the result expressed in calories by 252.

T_0 = temperature of shale at start of retorting (usually atmospheric temperature) in degrees C.

T_1 = temperature in degrees C. at which oil is first produced.

T_2 = temperature in degrees C. at end of distillation.

C' = average specific heat of fresh shale, between T_0 and T_1. (Here taken as 0.265).

C'' = average specific heat of oil vapors produced, between T_1 and T_2. (Here taken as 0.6.)

C''' = average specific heat of steam produced, between T_1 and T_2. (Here taken as 0.47.)

C'''' = average specific heat of gas produced, between T_1 and T_2. (Here taken as 0.35.)

C''''' = average specific heat of spent shale produced, between T_1 and T_2. (Here taken as 0.225.)

r' = latent heat of vaporization of oil produced. (Here taken as 100.)

r'' = latent heat of vaporization of water produced. (540 calories per gram.)

O = weight of oil produced in pounds.
W = weight of water produced in pounds.
G = weight of gas produced in pounds.
R = weight of spent shale (shale residue) in pounds.

The following calculation is shown as an example, in which values for shale No. 3, Table X, are used. (For a summarized result on all the ideal shales considered, see Table XIV.)

The shale yielded at the rate of 375 pounds of oil, 41.7 pounds of water, 1,383 pounds of spent shale, and 200 pounds of gas, determined by difference, all per ton of shale. The unit considered here is one ton.

Substituting in the formula shown:

$$454 \left\{ 2000 \left[(205 - 15)\ 0.265 \right] + 375 \left[100 + (482 - 205)\ 0.6 \right] + 41.7 \left[540 + (482 - 205)\ 0.47 \right] + 200 \left[(482 - 205)\ 0.35 \right] + 1383 \left[(482 - 205)\ 0.225 \right] \right\} = 149{,}272{,}000 \text{ calories or } 593{,}000 \text{ B.T.U.}$$

In other words, the dry distillation of one ton of the shale under consideration, to 482° C. (900° F.), and recovering the quantity of products above set forth, would require 149,272,000 calories or 593,000 B.T.U. of heat, if the retort were 100 per cent thermally efficient. Of course 100 per cent efficiency is never obtained, and therefore the above figure must, in practice, be multiplied by a factor based on the efficiency of whatever retort is used.

COMPARISON OF CALCULATED AND EXPERIMENTALLY DETERMINED HEAT REQUIREMENTS.

It is possible to apply the formula given to an experiment in which the actual amount of heat used in distilling an oil-shale was determined. Mr. Arthur J. Franks, of Golden, Colorado, working entirely independently of the authors, and without knowledge of the theoretical work being undertaken by the latter, has, by means of electrical measurements, roughly determined the amount of heat necessary to distil one ton of oil-shale. With his permission his experimental results and comments have been included:

Retorting data:

Weight of shale distilled, 10 pounds.
Average temperature inside retort wall, 535° C.
Time of retorting, 2 hours (7200 seconds).
Estimated thermal efficiency of retort, 75 to 85 per cent.

Heat measurements:

Heat, electrical.
Average voltage during test, 20.
Average amperage during test, 30.
Total watt seconds (20 × 30 × 7200) = 4,320,000.

One watt second = 0.2389 small calories.

4,320,000 × 0.2389 = 1,033,000 calories for 10 pounds of shale, or 206,600,000 gram calories per 2,000 pounds, or 820,000 B.T.U. per ton of shale.

Products obtained per ton of shale retorted:

Oil, specific gravity assumed 0.900 at 15.56° C., 47.76 gallons or 357.9 pounds.

Water, specific gravity assumed 1.000, 7.14 gallons or 59.5 pounds.

Gas, assumed specific gravity 1.24 (air = 1.0), (gas contains 10.9 per cent CO_2), 3,260 cubic feet or 326 pounds.

Spent shale, 1256.6 pounds.

Substituting the above weights in the formula given:

$$454 \{ 2000 [(205 - 15)\ 0.265] + 537.9 [100 + (535 - 205)\ 0.6] + 59.5 [540 + (535 - 205)\ 0.47] + 326 [(535 - 205)\ 0.35] + 1256.6 [(535 - 205)\ 0.225] \} = 170{,}064{,}000 \text{ calories or } 677{,}000 \text{ B. T. U.}$$

It will be recalled that Mr. Frank's experiment indicated that 820,000 B.T.U.'s were required to retort a ton of the shale experimented with, and he estimated that the thermal efficiency of his retort was from 75 to 85 per cent. Assuming the above calculated value represents the heat required at 100 per cent retort efficiency, Mr. Frank's retort was 82.56 per cent efficient.

Although this close agreement does not constitute a general proof of the reliability of the formula, or even an absolute proof for this specific case, it would seem to argue strongly in favor of the applicability of the formula for at least approximations until such time as more complete data allows the development of a more exact expression.

It is regretted that similar experimental data on shales of lower and higher oil yields are not obtainable to furnish bases for other confirmatory calculations, and the authors will greatly appreciate any authoritative data which may be used to test the accuracy of the formula, or to reduce the errors and assumptions known to be involved therein.

CALCULATION OF HEAT AVAILABLE FROM SHALE GAS AND SPENT SHALE.

From considerations developed in the paper on "Fuel Values of Shale and Shale Products," pages 13 to 21, the approximate heating value of each of the ideal shales was calculated by multiplying the assay yield in gallons of oil per ton by the factor 106.6 to arrive at B.T.U. value per pound, and by 2000 × 106.6 for B.T.U. value per ton. The results of these calculations are shown in Table XI.

From further considerations developed in the above mentioned paper, the percentage of heat found in each product was estimated, and also the heat per unit of each product. (See Table IV, page 18, and discussion, pages 15 and 16.) Some slight adjustment by the method of trial and error yielded percentages which, upon being applied, gave heat values to units that checked within reasonable limits those determined experimentally. By applying these percentages to the original heat values, the results presented in Tables XII and XIII were obtained. These figures indicate the amount of heat available in the products of retorting one ton of the ideal shales under discussion.

THERMAL EFFICIENCIES OF RETORTS NECESSARY TO RETORT OIL-SHALES OF DIFFERENT RICHNESS.

By applying the formula given on page 53 it is possible to determine the amount of heat required to retort the ideal shales discussed. Table XIV shows the results of such determinations, considering that the retorting is done in retorts of different heating efficiencies.

Now having obtained figures approximating the total heat necessary for retorting the shales (Table XIV), and also the heat which may be obtained from the spent shale or shale gas, or both, (Table XIII), there may be calculated the absolute thermal efficiency necessary for a retort which is to handle any particular shale, by dry distillation, by burning either the shale gas, or shale residue, or both, with no additional fuel. Results of such calculations for the ideal shales considered are given in Table XV.

Both the fuel necessary to retort and that recoverable from the shale depend, of course, on the maximum temperature to which the shale is raised. Therefore results of two sets of calculations, one using a low and the other a high final retorting temperature, are included in Tables XIV and XV.

All calculations are slide-rule estimations and therefore correct to the third significant figure.

TABLE X.

COMPOSITION BY WEIGHT OF ONE TON OF SHALE.[1]

Shale No.	Fresh shale Lbs.	Oil[2] Lbs.	Water Lbs.	Gas[3] Lbs.	Residue Lbs.	Assay		
						Oil Gals. per ton	Water Gals. per ton	Gas Cu ft. per ton
1	2000	187.5	41.70	125	1646.8	25.0	5.0	2500
2	2000	375.0	41.70	200	1383.3	50.0	5.0	4000
3	2000	562.5	41.70	250	1146.8	75.0	5.0	5000
4	2000	750.0	41.70	250	958.3	100.0	5.0	5000

[1] Ideal assumed shales (see page 52).

[2] Specific gravity of oil assumed as 0.900.

[3] Specific gravity of gas assumed as 0.656 (air = 1); 1 cu. ft. gas weighs 0.05 lbs.

TABLE XI.

TOTAL HEATING VALUE OF OIL-SHALES OF VARYING RICHNESS.

Shale No.[1]	Richness of shale gals. oil per ton	Factor[2]	B. T. U. per lb.	B. T. U. per ton.
1	25	106.6	2,665	5,330,000
2	50	106.6	5,330	10,660,000
3	75	106.6	7,995	15,990,000
4	100	106.6	10,660	21,320,000

[1] Numbers refer to Table X.

[2] See page 15.

TABLE XII.

HEAT VALUE OF PRODUCTS ON ONE TON OF OIL-SHALES OF DIFFERENT RICHNESS.

Shale No.[1]	Gals. oil per ton	Residue		Gas		Oil	
		Total B. T. U.	B. T. U. per lb.	Total B. T. U.	B. T. U. per cu. ft.	Total B. T. U.	B. T. U. per lb.
1	25	995,000	604	818,000	327	3,465,000	18,500
2	50	1,990,000	1,438	1,636,000	408	6,930,000	18,500
3	75	2,399,000	2,095	2,559,000	512	10,395,000	18,500
4	100	2,342,000	2,440	3,410,000	682	13,860,000	18,500

[1] Numbers refer to Table X.

TABLE XIII.

HEAT RECOVERABLE IN PRODUCTS OF OIL-SHALES OF VARYING RICHNESS.

Shale No.[1]	Richness of shale Gals. oil per ton	Heat in one ton of original shale in 1000 B. T. U.'s	Heat in all products recovered from one ton of shale in 1000 B. T. U.'s	Heat in recovered products from one ton of shale in 1000 B. T. U.'s				
				Oil	Gas	Spent shale	Oil and gas	Spent shale and gas
1	25	5,330	5,278	3,465	818	995	4,283	1,813
2	50	10,660	10,556	6,930	1,636	1,990	8,566	3,626
3	75	15,990	15,353	10,395	2,559	2,399	12,954	4,958
4	100	21,320	19,612	13,860	3,410	2,342	17,270	5,752

[1] Numbers refer to Table X.

TABLE XIV.

HEAT REQUIRED TO RETORT OIL-SHALES OF VARYING RICHNESS.

Shale No.[1]	Retort efficiency per cent				Retort efficiency per cent			
	100	50	30	20	100	50	30	20
	Heat required at low temperature, 482° C. (900° F.) in 1000 B. T. U.'s				Heat required at high temperature, 925° C. (1700° F.) in 1000 B. T. U.'s			
1	518	1,036	1,727	2,590	956	1,912	3,187	4,780
2	593	1,186	1,976	2,965	1,091	2,182	3,637	5,455
3	663	1,326	2,210	3,315	1,220	2,440	4,067	6,100
4	725	1,450	2,416	3,625	1,350	2,700	4,500	6,750

[1] Numbers refer to Table X.

TABLE XV.

NECESSARY THERMAL EFFICIENCIES OF RETORTS.

If spent shale and shale gas is the only fuel used.

	At low temperature 482° C. (900° F.)			At high temperature 925° C. (1700° F.)		
Richness of shale; gals. oil per ton	Heat necessary to retort one ton of shale in 1000 B. T. U.'s	Heating value of spent shale and gas from one ton shale; in 1000 B. T. U.'s	Necessary thermal efficiency of retort; per cent	Heat necessary to retort one ton of shale in 1000 B. T. U.'s	Heating value of spent shale and gas from one ton shale in 1000 B. T. U.'s	Necessary thermal efficiency of retort; per cent
25	518	1,813	28.55	956	1,813	52.70
50	593	3,626	16.35	1,091	3,626	30.05
75	663	4,958	13.38	1,220	4,958	24.60
100	725	5,752	12.59	1,350	5,752	23.45
If shale gas is the only fuel used.						
25	518	818	63.30	956	818	Over 100 %
50	593	1,636	35.25	1,091	1,636	66.80
75	663	2,559	25.90	1,220	2,559	47.70
100	725	3,410	21.25	1,350	3,410	39.60

CONVENIENT FACTORS FOR USE IN OIL SHALE CALCULATIONS.

The purpose of this paper is to present factors frequently used in making oil-shale calculations, in a form which will permit their use by the field operator. The factors presented are for purposes of arriving at close approximations.

All the figures in Table XVI are taken from Mark's "Mechanical Engineer's Handbook." The table showing effect of altitude on atmospheric pressure (Table XXV) is taken from "Metallurgists and Chemists Handbook," by Liddell. The other tables and formulae are those used by the authors in the course of calculations made by them. They are in general merely applications of well known principles to specific cases. The physical data for shales in Table XVII are those determined by the authors, most of them having been previously published.[1] Considerable care should be used in applying them as they were determined only for shales of a certain average richness from the Green River formation in Colorado. It is believed, however, that they may have rather wide application.

TABLE XVI.

FREQUENTLY USED EQUIVALENTS.

Length.

1 centimeter = 0.3937 inches.
1 meter = 3.281 feet.
1 meter = 1.0936 yards.
1 inch = 2.54 centimeters. (cm.)
1 foot = 0.3048 meters. (m.)
1 yard = 0.9144 meters. (m.)

Areas.

1 acre = 43,560 square feet.
640 acres = 1 square mile.

Volumes.

1 cubic inch = 16.39 cubic centimeters. (cc.)
1 cubic foot = 1728 cubic inches. (cu. in.)
1 cubic foot = 28,352 cubic centimeters. (cc.)
1 gallon = 231 cubic inches. (cu. in.)
1 gallon = 3785 cubic centimeters. (cc.)
1 gallon = 0.1357 cubic feet. (cu. ft.)
1 gallon = 0.004951 cubic yards. (cu. yds.)
1000 cubic centimeters = 1.0 liter. (l.)
1 liter = 0.03531 cubic feet. (cu. ft.)
1 cubic meter = 35.3 cubic feet. (cu. ft.)

Mass.

1 gram = weight of 1 cubic centimeter of pure water at 4° C.
28.35 grams (gm.) = 1 ounce. (oz.)
453.6 grams = 1 pound (lb.) = 16 ounces.
1000 grams = 1 kilogram (kg.) = 2.205 pounds.

Miscellaneous.

1 gallon of water (specific gravity = 1.0) = 8.328 pounds = 3780 grams.
1 cubic foot of water (at 40° C.) = 62.428 pounds.
1 cubic foot of water (at 100° C.) = 59.830 pounds.
1 pound of water. = 0.12 gallons.
1 pound of oil (specific gravity 0.9) = 0.1334 gallons.
1 barrel = 42 gallons = 350 pounds of liquid of specific gravity 1.000[1].
1 ton of oil (specific gravity 0.9) = 5.72 barels.
1 barrel of liquid = 5.6154 cubic feet.

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[1] Gavin, M. J., and Sharp, L. H., Some physical and chemical data on Colorado oil-shale, Bureau of Mines, Reports of Investigations, Serial No. 2152, August, 1920, 8 pp.

[1] To calculate pounds per barrel of any other liquid, multiply 350 by specific gravity of liquid.

TABLE XVII.

SOME CONSTANTS FOR SHALE AND SHALE PRODUCTS.

	Fresh shale	Spent shale	Shale oil	Shale gas[1]	Steam
Specific heat	0.265[2]	0.225[3]	0.5—0.6	0.35[4]	0.47
Calories per gram	Assay in gallons per ton x 59.25	Approximately 0.2319 x heat value of fresh shale from which it was derived.	10,270		
B. T. U. per pound	Assay in gallons per ton x 106.6	Approximately 0.2319 x heat value of fresh shale from which it was derived.	18,500	Varies from 300 to 600 B. T. U. per cu. ft. at 0°C., 760 mm.	
Latent heat of vaporization			100	Water 540	

[1] Produced from shale by dry destructive distillation.

[2] Colorado oil-shale yielding 42 gallons of oil per ton.

[3] Residue from Colorado oil-shale yielding 42 gallons of oil per ton.

[4] Approximately.

TABLE XVIII.

HEAT EQUIVALENTS.

The **calorie (small calorie or gram calorie)** is the quantity of heat required to raise the temperature of one gram of pure water from 4° to 5° Centigrade. The **mean calorie,** commonly used by engineers, and in this paper, is 1/100 of the quantity of heat required to raise the temperature of 1 gram of pure water from 0° to 100° Centigrade. It is nearly the same as the amount of heat required to raise the temperature of 1 gram of pure water from 17° to 18° Centigrade.

The **large Calorie (kilogram Calorie)** is 1000 times the small calorie, in whatever form the small calorie may be expressed.

The **British Thermal Unit** (B. T. U.) is the quantity of heat required to raise the temperature of 1 pound of pure water one degree Fahrenheit, at 39.1° F., the temperature of the maximum density of water. The **Mean British Thermal Unit** is 1/180 of the quantity of heat required to raise the temperature of one pound of pure water from 32° to 212° Fahrenheit. It is the term commonly used in American engineering practice.

1 B. T. U.=252 calories=0.252 kilogram Calories.
1 kilogram Calorie=3.968 B. T. U.'s.
To change calories per gram to B. T. U. per pound, multiply by 1.8 (9/5).
To change B. T. U. per pound to calories per gram, divide by 1.8 (or multiply by 5/9).

TABLE XIX.

TEMPERATURES.

Fahrenheit scale (F.): Freezing point of water=32°; boiling point of water=212°.

Centigrade scale (C.): Freezing point of water=0°; boiling point of water=100°.

Absolute scale (A.): Freezing point of water=273°; boiling point of water=373°.[1]

To change temperature in °F. to °C.: (F. reading—32) × 5/9=°C.
To change temperature in °C. to °F.: (C. reading × 9/5)+32=°F.
To change temperature in °C. to °A.. C. reading+273=°A.
To change temperature in °A to °C.: A. reading—273=°C.
To change temperature in °F. to °A.: convert to °C. then apply above.
To change temperature in °A. to °F.: convert to °C. then apply above.

[1] Absolute temperatures are sometimes expressed in Fahrenheit units instead of Centigrade units. Add 459° to Fahrenheit reading for this purpose.

TABLE XX.

WEIGHT OF SHALE.

(For approximations only.)

Specific gravity of shale.	Weight per cubic foot Pounds.	Weight per cubic foot Tons.	Weight per acre per foot of thickness.
1.5	93.7	.04685	2040 tons
1.6	99.9	.04999	2176 tons
1.7	106.1	.05305	2310 tons
1.8	112.3	.05615	2445 tons
1.9	118.7	.05935	2583 tons
2.0	125.0	.06250	2721 tons
2.1	131.1	.06555	2858 tons
2.2	137.3	.06865	2990 tons
2.3	143.6	.07180	3124 tons
2.4	150.0	.07500	3265 tons
2.5	156.3	.07815	3405 tons

Formula: 1 cubic foot = 28,353 cubic centimeters.

(1) $\frac{\text{Specific gravity} \times 28,353}{454}$ or (specific gravity × 62.5) = pounds per cubic foot.

(2) $\frac{\text{Pounds per cubic foot}}{2000}$ = tons per cubic foot.

(3) Tons per cubic foot × 43,560 = tons per acre per foot of thickness.

Weight of shale in place is much greater per cubic foot than when mined.

Weight per cubic foot mined varies from 0.42 to 0.5 of its weight in place according to the size to which it is crushed. The finer the shale is crushed the greater its weight per cubic foot.

One cubic foot in place when mined will yield $\frac{1}{0.42}$ to $\frac{1}{0.5}$, i. e., 2.38 to 2.0 cubic feet.

TABLE XXI.[1]

PETROLEUM OIL TABLE FOR CONVERTING SPECIFIC GRAVITY TO BAUME DEGREES.

Specific gravity 60°/60°F.	Degrees Baume (Modulus 140)	Pounds per gallon	Gallons per pound
0.600	103.33	4.993	0.2003
.610	99.51	5.076	.1970
.620	95.81	5.160	.1938
.630	92.22	5.243	.1907
.640	88.75	5.326	.1877
.650	85.38	5.410	.1848
.660	82.12	5.493	.1820
.670	78.96	5.577	.1793
.680	75.88	5.660	.1767
.690	72.90	5.743	.1741
.700	70.00	5.827	.1716
.710	67.18	5.910	.1692
.720	64.44	5.994	.1668
.730	61.78	6.077	.1646
.740	59.19	6.160	.1623
.750	56.67	6.244	.1602
.760	54.21	6.327	.1580
.770	51.82	6.410	.1560
.780	49.49	6.494	.1540
.790	47.22	6.577	.1520
.800	45.00	6.661	.1501
.810	42.84	6.744	.1483
.820	40.73	6.827	.1465
.830	38.68	6.911	.1447
.840	36.67	6.994	.1430
.850	34.71	7.078	.1413
.860	32.79	7.161	.1396

PETROLEUM OIL TABLE FOR CONVERTING SPECIFIC GRAVITY TO BAUME DEGREES—Continued.

Specific gravity 60°/60° F.	Degrees Baume (Modulus 140)	Pounds per gallon	Gallons per pound
.870	30.92	7.244	.1380
.880	29.09	7.328	.1365
.890	27.30	7.411	.1349
.900	25.56	7.494	.1334
.910	23.85	7.578	.1320
.920	22.17	7.661	.1305
.930	20.54	7.745	.1291
.940	18.94	7.828	.1278
.950	17.37	7.911	.1264
.960	15.83	7.995	.1251
.970	14.33	8.078	.1238
.980	12.86	8.162	.1225
.990	11.41	8.245	.1213

[1]This and the following three tables are taken from United States Standard tables for Petroleum Oils, Circular 57, U. S. Bureau Standards, 1916, 64 pp.

TABLE XXII.

PETROLEUM OIL TABLE FOR CONVERTING BAUME DEGREES TO SPECIFIC GRAVITY.

Degrees Baume (Modulus 140)	Specific gravity 60°/60° F.	Pounds per gallon	Gallons per pound	Degrees Baume (Modulus 140)	Specific gravity 60°/60° F.	Pounds per gallon	Gallons per pound
10.0	1.0000	8.328	0.1201	55.0	0.7568	6.300	0.1587
11.0	.9929	8.269	.1209	56.0	.7527	6.266	.1596
12.0	.9859	8.211	.1218	57.0	.7487	6.233	.1604
13.0	.9790	8.153	.1227	58.0	.7447	6.199	.1613
14.0	.9722	8.096	.1235	59.0	.7407	6.166	.1622
15.0	.9655	8.041	.1244	60.0	.7368	6.134	.1630
16.0	.9589	7.986	.1252	61.0	.7330	6.102	.1639
17.0	.9524	7.931	.1261	62.0	.7292	6.070	.1647
18.0	.9459	7.877	.1270	63.0	.7254	6.038	.1656
19.0	.9396	7.825	.1278	64.0	.7216	6.007	.1665
20.0	.9333	7.772	.1287	65.0	.7179	5.976	.1673
21.0	.9272	7.721	.1295	66.0	.7143	5.946	.1682
22.0	.9211	7.671	.1304	67.0	.7107	5.916	.1690
23.0	.9150	7.620	.1313	68.0	.7071	5.886	.1699
24.0	.9091	7.570	.1321	69.0	.7035	5.856	.1708
25.0	.9032	7.522	.1330	70.0	.7000	5.827	.1716
26.0	.8974	7.473	.1338	71.0	.6965	5.798	.1725
27.0	.8917	7.425	.1347	72.0	.6931	5.769	.1733
28.0	.8861	7.378	.1355	73.0	.6897	5.741	.1742
29.0	.8805	7.332	.1364	74.0	.6863	5.712	.1751
30.0	.8750	7.286	.1373	75.0	.6829	5.685	.1759
31.0	.8696	7.241	.1381	76.0	.6796	5.657	.1768
32.0	.8642	7.196	.1390	77.0	.6763	5.629	.1776
33.0	.8589	7.152	.1398	78.0	.6731	5.602	.1785
34.0	.8537	7.108	.1407	79.0	.6699	5.576	.1793
35.0	.8485	7.065	.1415	80.0	.6667	5.549	.1802
36.0	.8434	7.022	.1424	81.0	.6635	5.522	.1811
37.0	.8383	6.980	.1433	82.0	.6604	5.497	.1819
38.0	.8333	6.939	.1441	83.0	.6573	5.471	.1828
39.0	.8284	6.898	.1450	84.0	.6542	5.445	.1837
40.0	.8235	6.857	.1459	85.0	.6512	5.420	.1845
41.0	.8187	6.817	.1467	86.0	.6482	5.395	.1854
42.0	.8140	6.777	.1476	87.0	.6452	5.370	.1862
43.0	.8092	6.738	.1484	88.0	.6422	5.345	.1871
44.0	.8046	6.699	.1493	89.0	.6393	5.320	.1880
45.0	.8000	6.661	.1501	90.0	.6364	5.296	.1888
46.0	.7955	6.623	.1510	91.0	.6335	5.272	.1897
47.0	.7910	6.586	.1518	92.0	.6306	5.248	.1905
48.0	.7865	6.548	.1527	93.0	.6278	5.225	.1914
49.0	.7821	6.511	.1536	94.0	.6250	5.201	.1923
50.0	.7778	6.476	.1544	95.0	.6222	5.178	.1931
51.0	.7735	6.440	.1553	96.0	.6195	5.155	.1940
52.0	.7692	6.404	.1562	97.0	.6167	5.132	.1949
53.0	.7650	6.369	.1570	98.0	.6140	5.110	.1957
54.0	.7609	6.334	.1579	99.0	.6114	5.088	.1966
55.0	.7568	6.300	.1587	100.0	.6087	5.066	.1974

TABLE XXIII.[1]

TEMPERATURE CORRECTIONS TO READINGS OF SPECIFIC GRAVITY HYDROMETERS IN AMERICAN PETROLEUM OILS AT VARIOUS TEMPERATURES.

(Standard at 60°/60° F.)

Observed temperature	OBSERVED SPECIFIC GRAVITY						
	0.650	0.700	0.750	0.800	0.850	0.900	0.950
	Subtract from observed specific gravity						
30	0.016	0.015	0.014	0.012	0.011	0.011	0.011
32	.015	.014	.013	.012	.011	.010	.010
34	.014	.013	.012	.011	.010	.010	.010
36	.013	.012	.011	.010	.009	.009	.009
38	.012	.011	.010	.009	.008	.008	.008
40	.0105	.0095	.0090	.0080	.0075	.0070	.0070
42	.0095	.0085	.0080	.0070	.0065	.0065	.0065
44	.0085	.0075	.0070	.0065	.0060	.0060	.0055
46	.0075	.0065	.0060	.0055	.0050	.0050	.0050
48	.0065	.0060	.0055	.0050	.0045	.0045	.0040
50	.0050	.0050	.0045	.0040	.0035	.0035	.0035
52	.0040	.0040	.0035	.0030	.0030	.0030	.0030
54	.0030	.0030	.0025	.0025	.0020	.0020	.0020
56	.0020	.0020	.0020	.0015	.0015	.0015	.0015
58	.0010	.0010	.0010	.0005	.0005	.0005	.0005
	Add to observed specific gravity						
60	.0000	.0000	.0000	.0000	.0000	.0000	.0000
62	.0010	.0010	.0010	.0005	.0005	.0005	....
64	.0020	.0020	.0015	.0015	.0015	.0015	....
66	.0030	.0030	.0025	.0025	.0020	.0020	....
68	.0040	.0040	.0035	.0030	.0030	.0030	....
70	.0050	.0050	.0045	.0040	.0040	.0035	....
72	.0060	.0055	.0050	.0045	.0045	.0040	....
74	.0070	.0065	.0060	.0055	.0050	.0050	....
76	.0080	.0075	.0070	.0065	.0060	.0055	....
78	.0090	.0085	.0080	.0070	.0065	.0065	....
80	.010	.009	.008	.008	.007	.007	....
82	.011	.010	.009	.008	.008	.007	....
84	.012	.011	.010	.009	.009	.008	....
86	.013	.012	.011	.010	.009	.009	....
88	.014	.013	.012	.011	.010	.010	....
90	.015	.014	.013	.012	.011	.010	....
92	.016	.015	.013	.012	.011	.011	....
94	.017	.016	.014	.013	.012	.012	....
96	.018	.016	.015	.014	.013	.013	....
98	.019	.017	.016	.015	.014	.013	....
100	.020	.018	.017	.015	.014	.014	....
102	.021	.019	.018	.016	.015	.015	....
104	.022	.020	.018	.017	.016	.015	....
106	.023	.021	.019	.017	.016	.016	....
108	.024	.022	.020	.018	.017	.017	....
110	.025	.023	.021	.019	.018	.017	....
112	.026	.024	.022	.020	.019	.018	....
114	.027	.025	.022	.020	.019	.019	....
116	.028	.026	.023	.021	.020	.019	....
118	.029	.026	.024	.022	.021	.020	....
120	.030	.027	.025	.023	.022	.021	....

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[1] It is not definitely known that the figures in this table can be applied to shale oils, as the coefficients of expansion of shale oils are not known at present. Oils produced from different shales or under different conditions from the same shale, may have different coefficients, but it is believed that the above figures will apply for fairly close approximations in most cases. It is possible that new tables must be worked out for shale oils.

TABLE XXIV.[1]

TEMPERATURE CORRECTIONS TO READINGS OF BAUME HYDROMETERS IN AMERICAN PETROLEUM OILS AT VARIOUS TEMPERATURES.

(Standard at 60° F.; modulus 140.)

Observed temperature deg. F.	OBSERVED DEGREES BAUME							
	20.0	30.0	40.0	50.0	60.0	70.0	80.0	90.0
	Add to observed degrees Baume							
30	1.7	2.0	2.4	3.0	3.7	4.3	5.0	5.7
32	1.6	1.9	2.3	2.8	3.4	4.0	4.7	5.3
34	1.5	1.8	2.1	2.6	3.1	3.7	4.3	4.9
36	1.4	1.6	2.0	2.4	2.9	3.4	4.0	4.6
38	1.3	1.5	1.8	2.2	2.6	3.1	3.6	4.2
40	1.2	1.4	1.6	2.0	2.4	2.8	3.2	3.8
42	1.1	1.2	1.5	1.8	2.2	2.5	2.9	3.4
44	.9	1.1	1.3	1.6	2.0	2.2	2.6	3.0
46	.8	.9	1.1	1.4	1.7	1.9	2.3	2.7
48	.7	.8	.9	1.2	1.4	1.6	2.0	2.3
50	.6	.7	.8	1.0	1.2	1.4	1.6	1.9
52	.5	.6	.7	.8	1.0	1.1	1.3	1.5
54	.3	.4	.5	.6	.8	.9	1.0	1.1
56	.2	.3	.3	.4	.5	.6	.6	.7
58	.1	.1	.1	.2	.3	.3	.3	.4
	Subtract from observed degrees Baume							
60	.0	.0	.0	.0	.0	.0	.0	.0
62	.1	.1	.1	.2	.2	.3	.3	.4
64	.2	.3	.3	.4	.4	.6	.6	.7
66	.3	.4	.5	.6	.7	.8	.9	1.0
68	.5	.6	.6	.7	.9	1.1	1.3	1.4
70	.6	.7	.8	.9	1.1	1.4	1.6	1.7
72	.7	.8	.9	1.1	1.3	1.6	1.9	2.1
74	.8	.9	1.1	1.3	1.6	1.8	2.2	2.5
76	.9	1.1	1.3	1.5	1.8	2.1	2.5	2.8
78	1.0	1.2	1.4	1.7	2.0	2.4	2.8	3.1
80	1.1	1.3	1.5	1.8	2.2	2.6	3.1	3.5
82	1.2	1.4	1.7	2.0	2.5	2.9	3.4	3.9
84	1.3	1.5	1.8	2.2	2.7	3.2	3.7	4.3
86	1.4	1.7	2.0	2.4	2.9	3.4	4.0	4.6
88	1.6	1.8	2.1	2.6	3.1	3.7	4.2	4.9
90	1.7	2.0	2.3	2.7	3.3	3.9	4.5	5.2
92	1.8	2.1	2.4	2.9	3.5	4.2	4.8	5.6
94	1.9	2.2	2.6	3.1	3.8	4.4	5.1	5.9
96	2.0	2.3	2.7	3.3	4.0	4.6	5.4	6.3
98	2.1	2.4	2.9	3.4	4.2	4.9	5.7	6.6
100	2.2	2.6	3.0	3.6	4.4	5.1	6.0	6.9
102	2.3	2.7	3.2	3.8	4.6	5.4	6.3	7.2
104	2.4	2.9	3.3	4.0	4.8	5.7	6.6	7.5
106	2.5	3.0	3.5	4.2	5.0	5.9	6.9	7.9
108	2.7	3.1	3.6	4.3	5.2	6.2	7.2	8.2
110	2.8	3.2	3.7	4.4	5.4	6.4	7.5	8.5
112	2.9	3.3	3.9	4.6	5.6	6.7	7.7	8.8
114	3.0	3.4	4.0	4.7	5.8	6.9	7.9	9.1
116	3.1	3.6	4.1	4.9	6.0	7.1	8.2	9.4
118	3.2	3.7	4.3	5.1	6.2	7.3	8.5	9.8
120	3.3	3.8	4.4	5.3	6.4	7.5	8.8	10.1

---

[1] It is not definitely known that the figures in this table can be applied to shale oils, as the coefficients of expansion of shale oils are not known at present. Oils produced from different shales or under different conditions from the same shale may have different coefficients, but it is believed that the above figures will apply for fairly close approximations in most cases. It is possible that new tables must be worked out for shale oils.

TABLE XXV.

RELATION BETWEEN ALTITUDE AND BAROMETRIC PRESSURE.[1]

Altitude in feet	Barometer in inches	Atmospheric pressure in lbs. per sq in.	Proportionate atmospheric density
0.00	30.0	14.72	1.00
500.0	29.5	14.45	0.98
1,000.0	28.9	14.18	0.96
1,500.0	28.4	13.94	0.94
2,000.0	27.9	13.69	0.93
2,500.0	27.4	13.45	0.91
3,000.0	26.9	13.20	0.89
4,000.0	26.0	12.75	0.86
5,000.0	25.1	12.30	0.83
6,000.0	24.2	11.85	0.80
7,000.0	23.3	11.44	0.77
8,000.0	22.5	11.04	0.75
9,000.0	21.7	10.65	0.73
10,000.0	20.9	10.26	0.70

[1] Liddell, D. M., Metallurgists and Chemists Handbook: 2d ed. 1918, p. 112.

TABLE XXVI.

FACTORS FOR USE IN CALCULATING RESULTS OF OIL SHALE ASSAYS.[1]

Weight of retort charge			Weight of retort charge		
Grams	Ounces	Factor	Grams	Ounces	Factor
1	2	3	1	2	3
10	.35	.042	310	10.94	1.294
20	.71	.083	320	11.30	1.335
30	1.06	.125	330	11.65	1.377
40	1.41	.167	340	12.00	1.419
50	1.76	.209	350	12.36	1.460
60	2.12	.250	360	12.71	1.502
70	2.47	.292	370	13.06	1.544
80	2.82	.334	380	13.41	1.586
90	3.18	.376	390	13.77	1.627
100	3.53	.417	400	14.12	1.669
110	3.88	.459	410	14.47	1.711
120	4.24	.501	420	14.83	1.753
130	4.59	.542	430	15.18	1.794
140	4.94	.584	440	15.53	1.836
150	5.29	.626	450	15.88	1.878
160	5.65	.668	460	16.24	1.919
170	6.00	.709	470	16.59	1.961
180	6.35	.751	480	16.94	2.003
190	6.71	.793	490	17.30	2.045
200	7.06	.835	500	17.65	2.086
210	7.41	.876	510	18.00	2.128
220	7.77	.918	520	18.36	2.170
230	8.12	.960	530	18.71	2.212
240	8.47	1.001	540	19.06	2.254
250	8.82	1.043	550	19.41	2.295
260	9.18	1.085	560	19.77	2.337
270	9.53	1.127	570	20.12	2.379
280	9.88	1.168	580	20.47	2.420
290	10.24	1.201	590	20.83	2.462
300	10.59	1.252	600	21.18	2.504

For any given weight of shale used (column 1 or 2), select the corresponding factor in column 3; divide the number of cubic centimeters of oil collected by this factor to convert into gallons of oil per ton of shale. For shale charges whose weights in grams are not even multiples of ten, it will be necessary to interpolate to obtain the proper factor.

[1] Prepared by L. C. Karrick, assistant oil-shale technologist, Bureau of Mines.

TABLE XXVII.

OTHER FACTORS FREQUENTLY USED IN MAKING OIL-SHALE ASSAY CALCULATIONS.

Imperial gallons per long ton to United States gallons per short ton	Multiply by 1.0716
Pounds per long ton to pounds per short ton	Multiply by 0.893
Per cent nitrogen to pounds amonium sulphate per short ton	Multiply by 92.4

1 Ton = 2,000 pounds = 907,185 grams.
1 Cubic foot = 28,377 cubic centimeters.

$$\text{Cubic feet of gas per ton} = 32.037 \times \frac{\text{cubic centimeters of gas obtained}}{\text{grams of shale retorted}}$$

$$\text{Gallons of oil per ton} = 239.66 \times \frac{\text{cubic centimeters of oil obtained}}{\text{grams of shale retorted}}$$