In Figure 1 the results of the evaporation test for lignite creosote specimen I are compared with results of a similar test done by Vaughan (*12*) on coal-tar creosote. The purpose of these tests was to obtain some idea of the probable permanence of the preservative. From Figure 1 lignite creosote may be expected to be less satisfactory than coal-tar creosote in this respect. However, volatility effects may be offset to a degree by a possible greater ease of penetration of the wood by lignite creosote during impregnation.

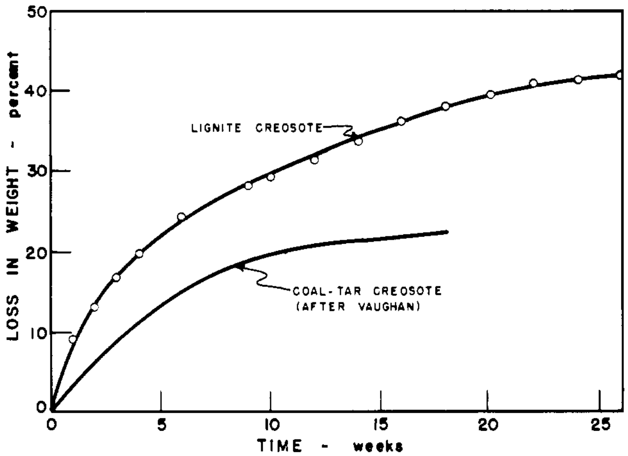

Figure 1. Evaporation curves of lignite and coal-tar creosotes

CONCLUSION

The proper evaluation of a wood preservative requires the use of service tests such as listed by Prostel (*8*) for lignite creosote. However, the toxicity values reported in the present study suggest that lignite creosote should prove to be a satisfactory wood preservative. Chlorination may enhance its usefulness, although means of eliminating sludge formation would have to be investigated.

ACKNOWLEDGMENT

The authors are indebted to the Saskatchewan Research Council for financial assistance.

LITERATURE CITED

(1) Abraham, H., "Asphalts and Allied Substances," 5th ed., Vol. 1, pp. 381–2, Van Nostrand, New York, 1945.
(2) Am. Soc. Testing Materials, ASTM Standards, Part II, p. 597, 1946.
(3) *Ibid.*, Part IV, p. 1041, 1955.
(4) Am. Wood-Preservers' Assoc., Manual of Recommended Practice, loose-leaf, A.W.-P.A., Washington, D.C., 1951.
(5) Baechler, R. H., *Proc. Am. Wood-Preservers' Assoc.* **43,** 94 (1947).
(6) Duncan, C. G., *Ibid.*, **48,** 99 (1952).
(7) Finholt, R. W., *Anal. Chem.* **23,** 1038 (1951).
(8) Prostel, E., *Proc. Am. Wood-Preservers' Assoc.* **45,** 62 (1949).
(9) Reid, W. T., U. S. Bur. Mines Inform. Circ. **7430,** 15, 22 (1948).
(10) Schmitz, H., others, *Ind. Eng. Chem., Anal. Ed.* **2,** 361 (1930).
(11) Van Groenou, H. B., Rischen, H. W. L., Van den Berge, J., "Wood Preservation during the Last 50 Years," pp. 179–194, 204–205, A. W. Sijthoff, Leiden, 1951.
(12) Vaughan, J. A., *Ind. Eng. Chem.* **41,** 526 (1949).

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Applicability of a Specific Gravity-Oil Yield Relationship to Green River Oil Shale

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A specific gravity–oil yield relationship developed by the Bureau of Mines, Department of the Interior, for oil shales of the Rifle, Colo., area (*6*) is applicable to oil shales from 10 locations in the Piceance Creek Basin of Colorado and the Uinta Basin of Utah.

Average values of weight of oil shale per unit volume and oil yield of oil shale per unit volume based on shale specific gravities estimated from this relationship showed error limits which were less than ± 2.6% of corresponding values based on determined shale specific gravities. For all the samples included in this study the standard deviation from determined oil yield values of oil yields estimated from determined specific gravities using the D-5 relationship was 2.0 gallons per ton. This datum indicates that 95% of estimated oil yields will fall within 4 gallons per ton of the determined values. Nine of the 10 cores chosen for this study were from the Piceance Creek Basin deposit in Garfield and Rio Blanco Counties, Colo. One core was from the Uinta Basin deposit in Uintah County, Utah. These cores afford the best coverage available at present for the Green River formation oil shales as a whole.

To compare oil shales of equivalent grade, sections of the cores having average oil yields of 25 gallons of oil per ton of shale were examined. These sections represent the Mahogany zone of the Green River formation, which is comprised of the richest oil shales in the formation and will probably be the first portion processed commercially to liquid fuels. The oil shales in this zone are commonly designated by their distance above and below a distinctive bed of analcite about 4 inches thick, known as the Mahogany marker. Geologic studies indicate that the Piceance Creek Basin and the Uinta Basin were once continuous, and the oil shales in both basins show the Mahogany marker and the Mahogany zone.

The relationship between specific gravity and oil yield is a consequence of the nature of oil shale. Specific gravity varies inversely and oil yield varies directly with the concentration of organic material in the shale. The relationship for oil shales of the Green River formation is not linear, because the percentage of organic material converted to oil and the specific gravity of the oil change with the amount of organic material. However, when the specific gravities of the constituents of oil shales from a deposit are uniform over the deposit, a relationship applicable to the entire deposit may be developed (*4, 5*). In the previous study the compositions of oil-shale samples from two locations in the Green River formation were shown to be uniform enough to allow development of an algebraic equation relating specific gravity to oil yield for each of two locations (*6*). The present study demonstrates the applicability of one of these relationships developed for test hole D-5 of the Rifle, Colo., area to nine other cores and, by implication,

Table I. Description of Cores and Their Selected Sections

Number on Map	Core: Source	Core: Name of well	Location: State	Location: County	Location: Sec.	Location: T.	Location: R.	Selected Section: No. of samples	Selected Section: Length, ft.[a]	Selected Section: Reference to marker, ft.[b]
1	Tell Ertl	Phil	Colo.	Rio Blanco	25	1S.	100W.	84	85.5	27.5 to −58
2	Weber Oil Co.	Marcedus No. 2	Colo.	Rio Blanco	30	3S.	98W.	56	170	85 to −85
3	Union Oil Co.	Betty	Colo.	Garfield	21	4S.	95W.	169	201	76 to −125
4	Bureau of Mines	Naval Hole E.	Colo.	Garfield	2	5S.	95W.	94	155	54 to −101
5	Pacific Oil Co.	Wheeler No. 1	Colo.	Garfield	12	5S.	98W.	105	111	35.4 to −75.6
6	Bureau of Mines	D-5	Colo.	Garfield	12	6S.	95W.	84	84	22 to −62
7	Pacific Oil Co.	Dragert No. 1	Colo.	Garfield	5	6S.	96W.	136	143	53 to −90
8	Pacific Oil Co.	Hardison No. 1	Colo.	Garfield	2	7S.	98W.	68	68	17 to −51
9	Continental Oil Co.	Corehole No. 2	Colo.	Garfield	3 & 4	7S.	99W.	41	71.8	35 to −36.8
10	Shell Oil Co.	Corehole No. 2	Utah	Uintah	17	11S.	24E.	56	56	24 to −32

[a] Maximum continuous length of core averaging 25 gallons of oil per ton.
[b] Positive values indicate top of section above the Mahogany marker, negative values indicate bottom of section below the marker.

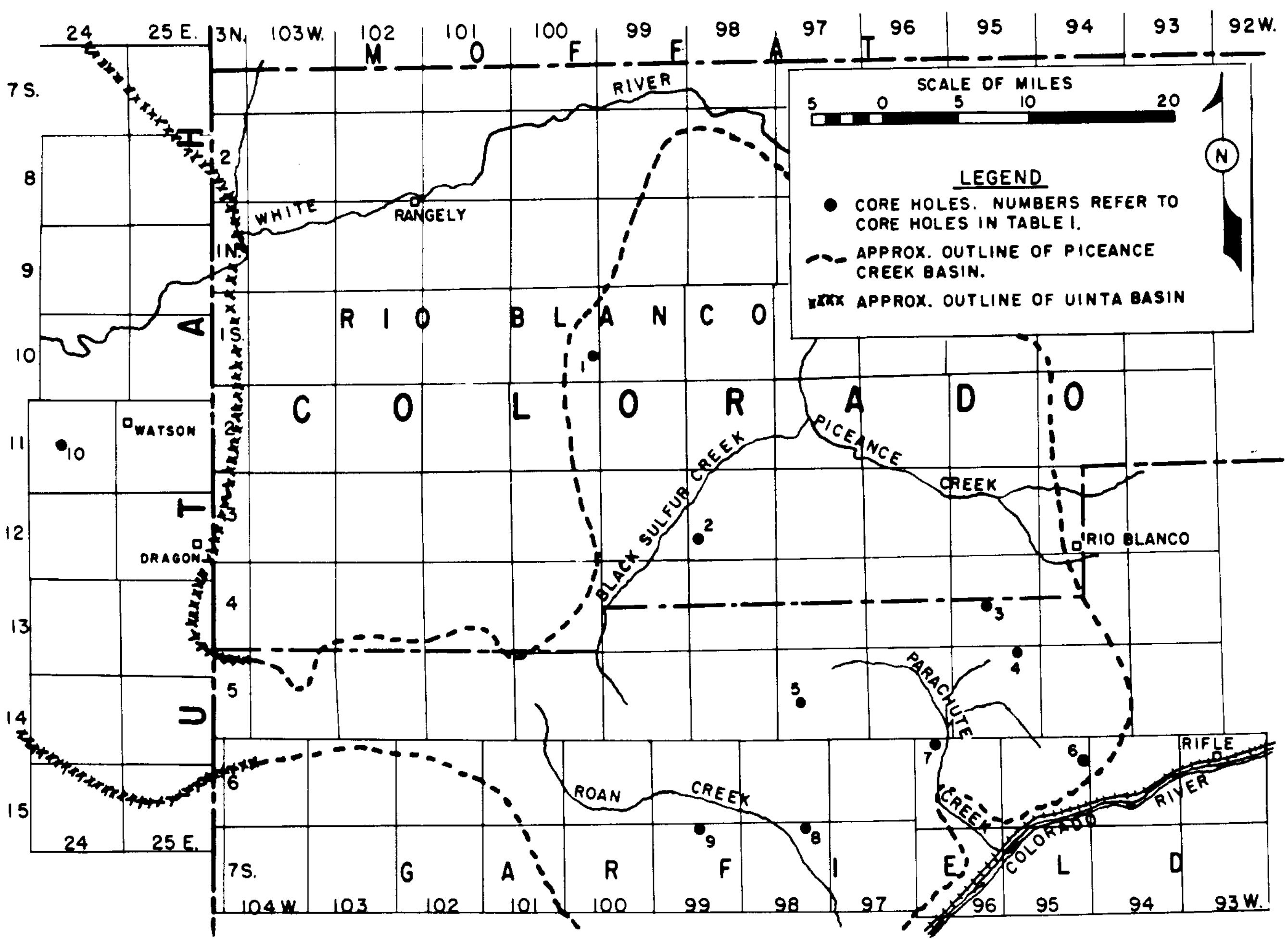

Figure 1. Location of core holes in Piceance Creek and Uinta Basins

to the Mahogany-zone shales of the Green River formation.

This specific gravity-oil yield relationship may be applied to Mahogany-zone shales for estimating oil yields from specific gravity of the samples (5). Estimation of oil-shale resources and reserves and evaluation of ore obtained by mining sequences of oil-shale beds are based on the oil yields per unit volume of the constituent beds. Analytically, the oil yields are determined on a weight basis and must be converted to a volume basis by considering the specific gravities of the oil shales. The D-5 relationship has been used extensively to make this conversion in the calculation of "demonstrated reserve" (7) as defined by Blondel and Lasky (3), who suggest that error limits of ±20% around the actual value for the ore in a deposit are acceptable. While other factors such as mining engineering limitations and economic considerations will affect the accuracy of the final estimate of reserves, this study was undertaken to provide a convenient method for minimizing the effect of variable specific gravity in oil-shale reserve calculations.

EXPERIMENTAL

Oil-Shale Samples. Descriptions of the 10 oil-shale cores and the selected 25 gallon-per-ton sections used in this study are given in Table I. All of the cores were from Mahogany-zone shales of the Green River formation in Colorado and Utah, from drilling sites within an area approximately 70 miles from east to west and 30 miles from north to south (see Figure 1). The 10 selected sections, totaling 1145.3 feet, were comprised of 893 individual samples of diamond-drilled core approximately 2 inches in diameter, which usually represented intervals of 1 foot. The sections represented the maximum continuous length of each core having an average oil yield of approximately 25 gallons per ton.

Methods of Analysis. The individual core samples were crushed to pass an 8-mesh-per-inch screen, air-dried, then analyzed for oil yield and for specific gravity at 60°/60° F. The oil yields were determined by the modified Fischer retort assay method (*8*). Specific gravities were determined by a procedure similar to those established by the American Society for Testing Materials for determining the specific gravities of soils (*2*) and the true specific gravities of burned refractory materials (*1*) modified by the use of *n*-heptane instead of water as the liquid medium.

Calculation Methods. The specific gravity–oil yield relationship tested in this study for applicability to Green River oil shales from different locations is expressed by the following equation:

$$y = 326.624 - 205.998x + 31.563x^2$$

where y is the oil yield in gallons per ton and x is the specific gravity at 60°/60°F. (*6*).

Based on this relationship, several specific properties for different grades of oil shale are given in Table II. The values in Table II differ slightly from the values published previously (*6*), owing to rounding the figures. The test for applicability of the relationship is based on comparison of average properties calculated from the estimated and the determined specific gravities of oil shales of equivalent grade that would be obtained by mining different deposits. The average oil yield of the ore obtained by mining a column 1 foot square through a sequence of oil-shale beds is calculated from the assayed oil yields, specific gravities, and footages of the individual core samples comprising the section. First, the oil yields of the samples, expressed in gallons of oil per ton of shale, are converted to gallons of oil per cubic foot, based on the specific gravities of the samples. The summation of the footages of the samples times their respective oil yields per cubic foot is the total oil yield of the column. This value, divided by the number of cubic feet in the column (numerically equal to the height of the column), gives the average oil yield in gallons of oil per cubic foot. This oil yield may also be expressed in gallons of oil per ton by considering the average weight of the column in pounds per cubic foot. The average weight per cubic foot of this column is obtained by a similar process of summing the footage of each sample times its weight per cubic foot and then dividing the total by the height of the column. To calculate the maximum continuous length of a section averaging 25 gallons of oil per ton of shale, as used in this study, the assayed oil yields and the related values given in Table II are used and samples are included in the summation until the average oil yield of the section is approximately 1.727 gallons of oil per cubic foot, which is equivalent to 25 gallons of oil per ton.

Table II. Volume-Weight-Oil Yield Relationships Based on D-5 Equation

Oil Yield by Assay, Gal./Ton	Sp. Gr. of Oil Shale, 60°/60°F.	Weight of Oil Shale per Unit Vol., Lb./Cu. Ft.	Vol. Oil Shale per Unit Weight, Cu. Ft./Ton	Oil Yield per Unit Vol., Gal./Cu. Ft.
0	2.714	169.08	11.829	0
2	2.660	165.72	12.069	0.166
4	2.610	162.60	12.300	0.325
6	2.564	159.74	12.520	0.479
8	2.520	157.00	12.739	0.628
10	2.478	154.38	12.955	0.772
12	2.439	151.95	13.162	0.912
14	2.400	149.52	13.376	1.047
16	2.364	147.28	13.580	1.178
18	2.330	145.16	13.778	1.306
20	2.297	143.10	13.976	1.431
22	2.264	141.05	14.179	1.552
24	2.233	139.12	14.376	1.669
25	2.218	138.18	14.474	1.727
26	2.203	137.25	14.572	1.784
28	2.173	135.38	14.773	1.895
30	2.145	133.63	14.967	2.004
32	2.117	131.89	15.164	2.110
34	2.089	130.14	15.368	2.212
36	2.063	128.52	15.562	2.313
38	2.037	126.91	15.759	2.411
40	2.012	125.35	15.955	2.507
42	1.986	123.73	16.164	2.598
44	1.961	122.17	16.371	2.688
46	1.937	120.68	16.573	2.776
48	1.914	119.24	16.773	2.862
50	1.890	117.75	16.985	2.944
52	1.867	116.31	17.195	3.024
54	1.845	114.94	17.400	3.103
56	1.823	113.57	17.610	3.180
58	1.801	112.20	17.825	3.254
60	1.779	110.83	18.046	3.325
62	1.758	109.52	18.262	3.395
64	1.737	108.22	18.481	3.463
66	1.717	106.97	18.697	3.530
68	1.696	105.66	18.929	3.592
70	1.676	104.41	19.155	3.654
72	1.656	103.17	19.385	3.714
74	1.637	101.99	19.610	3.774
76	1.618	100.80	19.841	3.830
78	1.598	99.56	20.088	3.883
80	1.579	98.37	20.331	3.935

RESULTS AND DISCUSSION

Average properties were calculated for each of the sections from the measured specific gravities and oil yields. These properties are compared in Table III with the same properties calculated for the same sections by using measured oil yields and specific gravities estimated from the D-5 relationship. The differences between the average weights calculated from the measured and from the estimated specific gravities range from 2.5 pounds per cubic foot below the estimated average to 1.1 above the estimated average. The difference between the average oil yields range from 0.030 gallon per cubic foot below the estimated average to 0.015 above and cover a range of 2.6% (+ 0.86 to − 1.76%). Because the specific gravity of the shale disappears in the calculations of the average oil yield in gallons per ton, these values have a maximum difference of only 0.2%, which is attributable to the rounding off of figures in the calculation process. Variations in the specific gravity-oil yield relationships for the different cores account for the differences in the other average properties.

To permit a direct comparison of the average properties of these core sections, each of the averages was adjusted to that for a shale having an average oil yield of 25.00 gallons per ton. These adjusted averages are given in Table IV in the order of their increasing size. Although the averages are higher for cores in the southern portion of the Piceance Creek Basin, there seems to be no definite trend to the variation. The values given by the D-5 equation for 25-gallon-per-ton oil shale are shown at the bottom of the table. These values are based on the equation calculated from data on 114 D-5 samples. Accordingly these values differ slightly from the values obtained for the 84 samples representing the 25-gallon-per-ton section of the D-5 core. In Table IV, the averages of the values for the properties of 25-gallon-per-ton oil shale from the 10 cores are 137.62 pounds per cubic foot and 1.720 gallons per cubic foot. The properties of the individual cores range within 2.6% of these averages and properties based on the D-5 equation are 0.41% higher than these averages.

Table V presents the standard deviations of the measured specific gravities from those estimated by the D-5 relationship. The standard deviation is a measure of the scatter or variation of points from the estimated value. The standard deviation values are increased by the differences between the specific gravity-oil yield relationships of the core D-5 and are not produced entirely by variability of the samples. Table V also gives the average difference of the measured gravities from the estimated gravities. This average difference was obtained by adding the deviations,

Table III. Average Properties of Selected Sections of Ten Cores

Number on Map	Core	Type of Data	Av. Oil Yield, Gal./Ton	Av. Weight, Lb./Cu. Ft.	Av. Oil Yield, Gal./Cu. Ft.
1	Phil	Determined	24.98	136.25	1.702
		Calculated[a]	24.98	138.19	1.726
2	Marcedus No. 2	Determined	25.21	135.52	1.708
		Calculated[a]	25.19	138.01	1.738
3	Betty	Determined	25.05	136.19	1.706
		Calculated[a]	25.04	138.14	1.729
4	Naval hole E	Determined	25.14	136.18	1.712
		Calculated[a]	25.09	138.10	1.732
5	Wheeler No. 1	Determined	25.06	138.54	1.736
		Calculated[a]	25.03	138.15	1.729
6	D-5	Determined	25.20	138.01	1.739
		Calculated[a]	25.16	138.02	1.736
7	Dragert No. 1	Determined	25.00	138.18	1.727
		Calculated[a]	25.00	138.18	1.727
8	Hardison No. 1	Determined	25.25	139.03	1.755
		Calculated[a]	25.23	137.95	1.740
9	Conoco corehole No. 2	Determined	24.98	138.06	1.724
		Calculated[a]	25.03	138.15	1.729
10	Shell corehole No. 2	Determined	24.89	138.17	1.720
		Calculated[a]	24.85	138.35	1.719

[a]Based on estimated specific gravities from D-5 relationship given in Table II.

Table IV. Properties of 25.00 Gallon-per-Ton Oil Shale from Ten Cores

Number on Map	Core	Av. Weight, Lb./Cu. Ft.	Av. Oil Yield, Gal./Cu. Ft.
2	Marcedus No. 2	136.08	1.701
1	Phil	136.20	1.703
3	Betty	136.32	1.704
4	Naval hole E	136.57	1.707
10	Shell corehole No. 2	137.88	1.724
9	Conoco corehole No. 2	138.01	1.725
7	Dragert No. 1	138.18	1.727
6	D-5 determined	138.55	1.732
5	Wheeler No. 1	138.70	1.734
8	Hardison No. 1	139.70	1.746
	Average	137.62	1.720
	D-5 equation	138.18	1.727

Table V. Standard Deviation and Average Difference of Measured and Estimated Specific Gravities for Ten Core Sections

Number on Map	Core	Average Difference[a]	Standard Deviation
1	Phil	−0.03615	0.0437
2	Marcedus No. 2	−0.03689	0.0407
3	Betty	−0.03220	0.0387
4	Naval hole E	−0.02773	0.0367
5	Wheeler No. 1	+0.00186	0.0206
6	D-5	−0.00061	0.0290
7	Dragert No. 1	−0.00385	0.0231
8	Hardison No. 1	+0.01284	0.0210
9	Conoco corehole No. 2	−0.00346	0.0172
10	Shell corehole No. 2	−0.00271	0.0193

[a]In specific gravity units. Plus and minus signs indicate that measured specific gravities were more and less, respectively, than estimated specific gravities.

taking the sign or direction of deviation into account, and dividing the total deviation by the number of samples. It is a measure of the size and direction of the difference between the specific gravity-oil yield relationship of the core section and the D-5 relationship.

Confidence bands measured as the distance above and below the curve established by the D-5 equation and including 95% of the data points may be approximated as the product of t_{95} times the standard deviation of the data points from the curve (6). The value for t_{95} varies with the number of samples but it is approximately 2 for the number of samples considered here. These bands then will be about 0.058 specific gravity unit above and below the D-5 curve. At a specific gravity of 2.218, which corresponds to the 25-gallon-per-ton shale in Table I, these bands are at ± 2.6% of the specific gravity. The 95% confidence-band distances from the D-5 curve are larger than all of the average difference values for all of the cores. Consequently, the specific gravity-oil yield relationships for all 10 cores lie within these limits. Although individual data points may be outside these bands, average values for the cores or core sections yielding about 25 gallons of oil per ton will lie within ± 2.6% of the D-5 curve. For the 10 core sections included in this study, the error between the averages calculated from the measured and the estimated specific gravities covered a range of only 2.6% (−1.76 to + 0.86%). Such accuracy is acceptable for calculation of reserves.

The standard deviation and average difference values given in Table V also indicate the accuracy of estimates of oil yield from determined specific gravities using the D-5 relationship. For all of the 893 samples included in this study the standard deviation of these estimates was 2.0 gallons per ton and, consequently, the range including 95% of the data points was about ± 4.0 gallons per ton. For closer estimates the average deviation values for the particular location must be considered. These represent systematic errors resulting from the application of the D-5 relationship to shales from other locations. Their size may be computed from the data given in Table V by correcting the specific gravity of 25-gallon-per-ton shale given by the D-5 relationship (specific gravity = 2.218) appropriately for the size and sign of the average deviation and calculating the gallons per ton from the equation using the resulting specific gravity. The difference between 25.0 and this calculated value is the systematic error. These errors range in size from estimates 2.4 gallons per ton too high for Marcedus No. 2 down to no systematic error for the D-5 core. If this correction is applied by reducing each oil yield estimate for the Marcedus No. 2 core by 2.4 gallons per ton, the standard deviation of the estimates is reduced from 2.71 to 1.17 gallons per ton.

Accuracy of uncorrected oil yield estimates from specific gravity is acceptable for most applications. Accuracy of specific gravity estimates from oil yield is acceptable for calculation of oil-shale reserves or resources. Consequently, the D-5 specific gravity–oil yield relationship may be applied to oil shales in the areas of the Piceance Creek and Uinta basins covered by this study. It may also apply generally to all Green River oil shales, but this should be confirmed by performing similar tests on oil-shale deposits in other areas of Utah and Wyoming.

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LITERATURE CITED

(1) Am. Soc. Testing Materials, Designation C 135–47, Standards, Part 3, p. 739, 1955.
(2) *Ibid.*, Designation D 854–52, p. 1786, 1955.
(3) Blondel, F., Lasky, S. G., *Economic Geol.* **51**, 686–97 (1956).
(4) Englhardt, D. von, *Brennstoff-Chem.* **13**, 10 (1932).
(5) Frost, I. C., Stanfield, K. E., *Anal. Chem.* **22**, 491–2 (1950).
(6) Smith, J. W., *Ind. Eng. Chem.* **48**, 441 (1956).
(7) Stanfield, K. E., others, U. S. Bureau of Mines, Rept. Invest. **5321** (1957).
(8) Stanfield, K. E., Frost, I. C., *Ibid.*, **4477** (1949).

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Preparation and Properties of Some Organophosphorus Compounds

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The excellent properties of tributyl phosphate (TBP) as a solvent for the extraction of metal salts from aqueous solutions prompted a survey of other organophosphorus compounds in these laboratories. Tributyl phosphate contains the electronegative phosphoryl oxygen and it was of interest to observe the effect of changing the basicity of this oxygen by comparing phosphine oxides, R_3PO; phosphinates, $R_2(RO)PO$; and phosphonates $R(RO)_2PO$; with the phosphates $(RO)_3PO$. Differences in polarity as indicated by heats of solution have been demonstrated for several of these compounds by Kosolapoff and McCullough (*14*). Within any of these classes further modifications could be made by substitution in the alkyl groups. In addition to changing the solvent strength, this variation in structure should influence the solubilities, densities, viscosities, and vapor pressures. In the present discussion these properties are compared for a number of compounds of the above classes. A discussion of the extraction properties for uranium and plutonium has been published (*2*).

PREPARATION OF COMPOUNDS

The compounds studied are listed in Table I. As indicated, several of the phosphates and a few phosphonates were obtained from Victor Chemical Co., Virginia Carolina Chemical Corp., Monsanto Chemical Co., and Ohio-Apex Co. The remaining compounds were synthesized by the methods indicated. Many of these methods are discussed by Kosolapoff (*11*).

The final compounds were purified by distillation at reduced pressure. The compounds are hygroscopic in varying degrees and hence were kept in tightly stoppered bottles. Where not otherwise identified, the literature values listed in the table are those cited by Kosolapoff (*11*).

MEASUREMENTS

Boiling points reported are those obtained during purification. Density, refractive index, and viscosity were measured in the conventional manner. Water-free samples were used for all physical measurements, because hydrogen bonding, which results when water is added, markedly influences these properties. The viscosity of tributyl phosphate, for example, changes from 33.2 to 39.9 millipoises when the compound is saturated with water. Solubility was determined by the cloud point technique and must be considered an approximation. Solubility data for tributyl phosphate are an exception and were obtained with the phosphorus-32 labeled ester.

RESULTS AND DISCUSSION

Boiling Points. The boiling points observed during vacuum fractionation are listed in Table I, with literature values when available. The alkyl esters of phosphinic and phosphonic acid show a boiling point increase of about 9° per CH_2 group at 1 mm. of mercury pressure. The vapor pressure curve of tributyl phosphate given by Evans (*5*), $\log P_{mm.} = 8.5861 - 3206.5/° K.$, was used as a reference for this comparison. The phosphates, phosphonates, and phosphinates containing the same number of carbon atoms appear to have about the same boiling points. The drop in molecular weight on changing from phosphate to phosphinate is perhaps compensated by increased polarity. More refined measurements would probably show more differences between them. Kosolapoff (*12*) has pointed out

References to Table I:

A. Victor Chemical Works.
B. $(RO)_2POCl + R'OH + \text{pyridine} \longrightarrow (RO)_2P(O)OR' + \text{pyr} \cdot HCl$ (*11*, p. 224).
C. Tributyl phosphate has been obtained from several vendors. Physical properties have been well established in this and other laboratories.
D. Ohio-Apex, Inc.
E. $3ROH + POCl_3 + \text{pyridine} \longrightarrow (RO)_3PO + \text{pyr} \cdot HCl$ (*17*).
F. Michaelis–Arbuzov Reaction (*6*) $P(OR)_3 + R'X \longrightarrow (RO)_2P(O)R' + RX$ (*11*, p. 121).
G. Monsanto Chemical Co.
H. $(C_4H_9O)_2P(O)H + Na + RBr \longrightarrow RP(O)(OC_4H_9)_2 + NaBr$ (*9*).
I. $RMgBr + ClP(O)(OC_4H_9)_2 \longrightarrow RP(O)(OC_4H_9)_2$. Standard Grignard technique. Cleavage occurred when attempted with diethyl-chlorophosphate.
J. $\phi POCl_2 + 2\,ROH \longrightarrow (RO)_2P(O)\phi + 2HCl$ (*18*).
K. $CCl_4 + P(OC_2H_5)_3 \longrightarrow CCl_3P(O)(OC_2H_5)_2 + C_2H_5Cl$ (*10*).
L. $\phi C(O)Cl + P(OC_2H_5)_3 \longrightarrow \phi C(O)P(O)(OC_2H_5)_2 + C_2H_5Cl$ (*7*).
M. $RBr + Mg + (R'O)_2P(O)H \longrightarrow R_2P(O)H$
$R_2P(O)H + H_2O_2 \longrightarrow R_2P(O)OH + H_2O$
$R_2P(O)OH + PCl_5 \longrightarrow R_2P(O)Cl$
$R_2P(O)Cl + R'ONa \longrightarrow R_2P(O)OR'$ (*15*).
N. $POCl_3 + RBr + Mg \longrightarrow R_3PO$ (*8*).
O. $(BuO)_2P(O)H + OctBr + Mg \longrightarrow (Oct)_2P(O)H$ (*19*).
P. Virginia–Carolina Chemical Corp.

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