Vol. 27. No. 208. February 1941.

A TECHNICAL STUDY OF TRANSVAAL TORBANITE.*

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Section A. Introduction.

The author has taken the opportunity of carrying out a survey of Transvaal torbanite in a more systematized manner than has hitherto been attempted. Fortunately access to the requisite number of samples of varying character was readily available from the vast deposits of the South African Torbanite Mining and Refining Co., Ltd.

There are two main classes of Transvaal torbanite which have been considered by the author—viz., the richer-quality torbanite as generally obtained from the Troye or Carlis' Adits, and the poorer class as mostly obtained from the Giesecke Adit of the well-known Ermelo deposits. This differentiation is necessary in order to emphasize the wide variation in the composition of torbanites.

Table III (Section B—Experimental), giving analyses of a large number of samples of varying composition, has been compiled with a view to classification according to " fuel ratio "—*i.e.*, ratio of organic volatiles to fixed carbon.

Generally speaking, the grade of torbanite is related to its ash content.

Three classes of mineral matter may be included in the ash value of the torbanite :—

(*a*) Inherent mineral matter which is so intimately mixed with the torbanite as to preclude its separation by ordinary means.

(*b*) Adventitious mineral matter, such as pyrites, nodules, shale, etc., which is deposited and mixed with the torbanite in such a way that its separation by ordinary means is quite feasible.

(*c*) Adventitious mineral matter included in the torbanite substance but derived from bands of pure mineral matter at the working face.

The fact that the determination of ash in any sample is a measure of the total mineral matter present, but is not indicative of the different possible forms, prompted Hall [1] to investigate the possibility of differentiation between the adventitious and inherent mineral matters present in commercial coal samples. Similar considerations prompted the author to apply ore-dressing principles for investigating the technical possibility of improving the nature of raw torbanite intended for industrial retorting.

The first step was naturally to attempt a grading of torbanite by dry screening. In order to use the optimum size in subsequent work, the average size of the oily gels was determined by microscopic examination. The various fractions of the mineral were then separated according to gravity by float-and-sink methods, using appropriate liquid mediums. Finally a hydraulic classification examination, using different methods in order to investigate the possibility of commercial washing of the raw torbanite prior to retorting, was carried out.

According to definition,[2] oil shale yields oil on distillation, but not appreciably when extracted with the common organic solvents for petroleum.

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* Paper received April 1939.

Extraction by solvents offers the possibility of isolating material which, if not existing in exactly the same form as in the parent substance, may at least provide a clue as to the structure of the parent substance more surely than other methods of examination such as thermal decomposition or behaviour when treated with powerful reagents.

High-pressure solvent-extraction methods, with particular reference to coal, by Bone[3] and Fischer[4] are described in Bakes'[5] outstanding study, but little data have been published on the chemical nature of the extracts.

It has not yet been found possible to produce from any oil shale only such hydrocarbons as are desirable. For example, the operation cannot be controlled in such a way that only the required degree of decomposition can be secured and the retorting then stopped at that particular point. Some of the constituents of the oil are consequently objectionable, and must be removed during refining operations.

In order to obtain from torbanite the maximum yield of optimum-quality oil consistent with economics, there are several basic factors necessitating consideration. The theoretical best conditions would naturally require certain modifications to meet commercial considerations of cost and feasibility.

The primary variables in the retorting of torbanite are :—

(*a*) The rate of temperature rise.

(*b*) The maximum temperature of carbonization.

(*c*) The pressure under which retorting is conducted.

(*d*) The rate of removal of oil vapours and gas—*i.e.*, the duration of contact of vapours with the heated zone.

(*e*) The fineness of the shale being retorted.

Other factors include the method of heat transfer, the thickness of the charge, and the presence in the retort of different gases and vapours which may have a bearing on the quantity and quality of the final products.

In order to ascertain the optimum conditions for retorting in the author's own experiments, it was necessary to make a survey of the literature[6–13] on oil-shale carbonization to correlate results obtained under different working conditions, and then to select those which would most probably be suitable for the problem on hand.

An examination of the literature referred to tends to show that no very wide variations in the quality of oil can be expected at different rates of heating. At fast rates and also when steam is used there appears to be a larger proportion of heavy constituents in the oil. At slow rates the resulting oils contain a somewhat greater proportion of light fractions and generally have a higher degree of saturation. It appears that in practice, in order to obtain an economic balance between oil yield and quality, a medium rate of distillation may be preferable.

The specific heats of raw and spent shale are high—*i.e.*, 0·265 and 0·223, respectively, according to Gavin.[2] Arising from this, in conjunction with the fact that oil shales are very bad conductors, attention has to be specially directed to maximum distribution of heat to every particle, especially as the best results from rich torbanite are obtainable only at the lowest temperature consistent with adequate exhaustion of the material and a reasonable rate of throughput.

This can be accomplished by carbonizing small particles and by keeping them in a state of agitation. The action of breaking down the gels to form oil is a surface one, and the greater the surface exposed, therefore, the more easily will carbonization proceed. This indicates the advisability of using retorts, preferably of a horizontal or inclined type, so that the material might be spread out more easily than with vertical retorts. In the latter the charge is constantly disturbed either by rotation of the retort or by the use of paddles or other stirring arrangements.

The size of particles, which should be as small as possible, and the rate of agitation, which should be as high as possible, are both limited in practice by the necessity of keeping down dust formation to a minimum. Dust produced by the agitation of very fine particles is inevitably carried away to the condensers, causing inconvenience by choking up the outlet mains and then appearing in the condensed oil. For a suitably designed retort the dust content of the oil should never exceed 0·5 per cent. Dust removal, mechanically or by electrostatic precipitation, for example, is costly, and, on the whole, prevention is better than cure. One method to accomplish this would be to design a retort in which a high rate of removal of oil vapours is accompanied by a low velocity of gases, so that such dust as is carried over is induced to drop back within the carbonization chamber.

It is also necessary to investigate the optimum thickness of the charge in the retort in order to ascertain " the depth to which heat can penetrate without injury to the external walls of the retort by running them at too high a temperature." This has been found by Kurth [6] to be approximately 16 inches. It is stated that if this thickness is increased considerably, the additional time required for retorting tends to reduce the oil yield perceptibly.

Factors such as size of carbonization unit and the amount of shale retorted, rate of distillation, the use of external or internal heating, the maximum temperature of carbonization, and the pressure under which retorting is conducted, etc., all have a marked influence on the yield of products and on their character.

In order that comparable results should be obtained, it is undoubtedly desirable that a uniform technique be employed in assay work. The apparatus described by different workers show variations from simple laboratory 10-gram retorts to semi-commercial units.

For rapid assay work affording a reasonable degree of reliability, the standard coal assay apparatus of Gray-King[14] has been widely adopted by several investigators for low-temperature carbonization work. The method of Fischer [15] using an aluminium retort has also wide application. These tests should always be carried out under standard conditions of temperature, time, etc., for the sake of comparison. In both methods only 10–20 grams of material are carbonized, and consequently the yield of oil or tar is much too small for any detailed examination. For this reason also the progress of carbonization cannot be studied by such methods.

Petrick,[11] in his valuable study on South African oil shales, in addition to the Gray-King apparatus used a stationary vertical retort carbonizing approximately 1500 grams at 570–580° C.

For the author's own work on carbonization a 14-lb. horizontal rotary retort (described fully under Section B) was constructed with a view to optimum operation based on the foregoing principles. Sufficient oil could thus be obtained for further examination, the course of carbonization could be progressively followed, and it was possible to obtain other information, such as the effect of temperature of evolution on oil and gas composition. It is not possible to collect such valuable data in small test units.

Other experimental work described includes an examination of the ash obtained from the spent torbanite after carbonization.

As the chief aim nowadays in the treatment of crude oils is to produce the maximum amount of petrol from them, after the straight-run spirit has been distilled off, it is necessary to crack the heavier oils left behind in order to increase the overall yield of petrol.

No complete cracking tests on Transvaal torbanite oil have previously been published, and the author gratefully acknowledges his indebtedness to the South African Torbanite Mining and Refining Co., Ltd., for kind permission to include one particular cracking test in this paper.

SECTION B. EXPERIMENTAL.

I. *Physical Examination.*

(a) *Physical Characteristics and Microscopic Examination.*

Transvaal torbanite samples investigated by the author ranged in colour from dull brown to velvety-black; streaks were yellow or fawn-coloured, and fractures across the bedding conchoidal. The material was characteristically very fine-grained and free from grit.

Under the microscope, thin sections of torbanite revealed the presence of innumerable particles of fatty resinous matter scattered through the ground-mass of the mineral as small globules or irregular streaks. These ranged in colour from yellow or reddish-yellow for high-grade samples to dark brown or nearly black for deteriorated torbanite, and their abundance appeared to be a measure of the oil-yielding properties of the mineral.

In order to facilitate subsequent work, it was necessary to know the size of the kerogen gels,[16] as it was considered that the most favourable conditions of separation would prevail if the mineral were crushed to approximately the same size.

Ten torbanite slides, prepared from samples yielding high and low proportions of oil, were thus examined under the microscope. The sizes of individual gels were measured for each slide, from which the mean true size was calculated. This is given in Table I, together with the maximum and minimum variation obtained. The corresponding sizes of I.M.M. screens are also recorded.

TABLE I.

Size of Kerogen Gels.

	Mm.	I.M.M. mesh.
Mean true size . . .	0·14	90
Minimum size . . .	0·07	180
Maximum size . . .	0·20	60

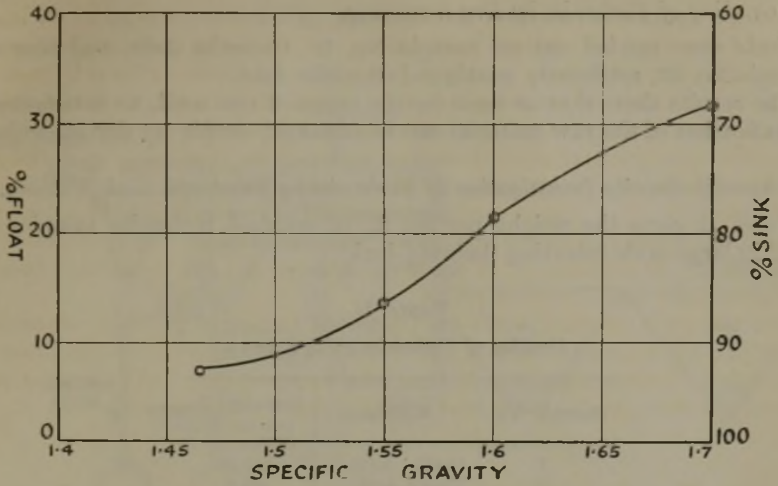

Fig. 1.

FLOAT AND SINK CURVE FOR SECOND GRADE TORBANITE.

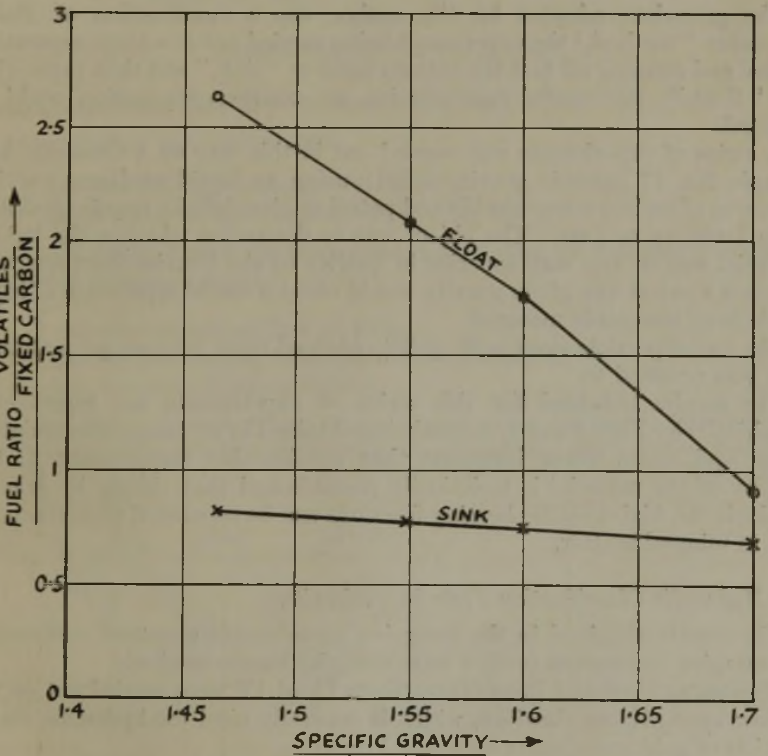

Fig. 2.

FUEL RATIOS OF SECOND GRADE TORBANITE.

(b) *Grading of Torbanite by Dry Screening.*

Tests were carried out on Sample No. 12, Giesecke Adit, and also on Sample No. 23, artificially weathered Giesecke Adit.

The results show that at least for the range of size used, no satisfactory classification of the raw material can be obtained simply by dry screening.

(c) *Specific Gravity Investigation of Torbanite by Float-and-Sink Methods.*

Table II gives the weight per cu. ft. of crushed torbanite samples as used in large-scale retorting (below ⅜-inch).

TABLE II.

Density of Torbanite (Size ⅜-inch).

Sample No.	Source.	Density, lb. per cu. ft.
7	Carlis' Adit	45·2
5	Troye Adit	49·6
17	Giesecke Adit	60·8

The procedure adopted by the author was a modification of Hall's "breaker" method,[1] the experiment being carried out in a large separating funnel and running off first the bottom layer or "sink," and then recovering the "float." By careful manipulation an excellent separation could be effected.

A series of experiments was carried out in this way on a Giesecke Adit Sample No. 17 (specific gravity 1·601), using as liquid mediums varying mixtures of carbon tetrachloride and petrol to give definite specific gravities from 1·465 up to 1·60. The object was to determine whether the lighter material was in any way superior in quality to the heavier fractions, and whether a cut at any given gravity would effect a useful separation of high-grade from low-grade material.

The use of particle size (— 60 + 90) obtained from microscopic examination was resorted to.

The results obtained for this series of experiments are represented graphically by Figs. 1 and 2, curves being obtained in all cases for cumulative floats and sinks. These illustrate that considerable impovement in the quality of the material is technically possible and that simply by gravity methods the high-volatile-bearing fractions can be separated from fractions rich in mineral matter.

(d) *Hydraulic Classification Tests on Torbanite.*

The results obtained in the foregoing experiments appeared sufficiently encouraging to warrant further tests using hydraulic methods.

Apparatus Used and Procedure.—Tests I and IV were carried out on the Callow upward-flow classifier, which is generally used for hydraulic classification of gold and similar ores (see Fig. 3).

It appears that the particle size used for classification has an important bearing on the separation of the different qualities of raw material.

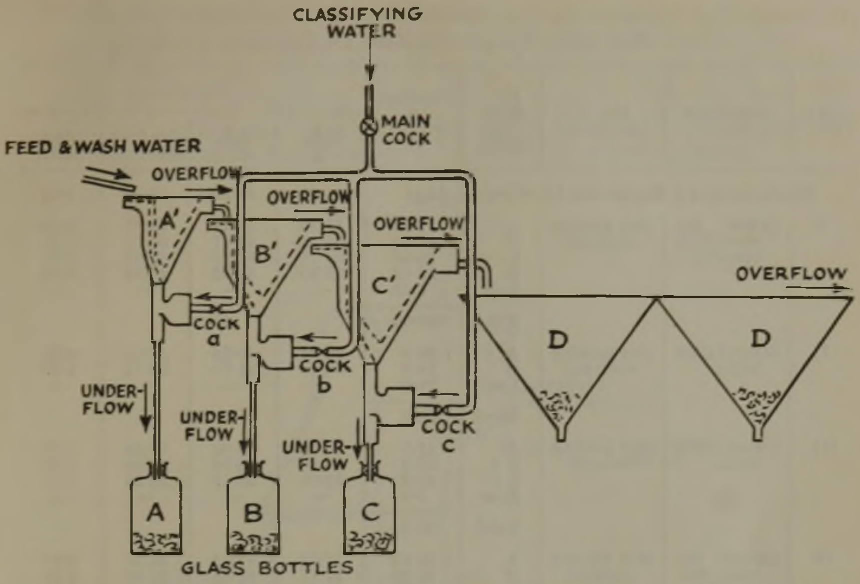

FIG. 3.

CALLOW UPWARD-FLOW CLASSIFIER.

Further Hydraulic Classification Tests using a Long Tube Classifier.

As a result of the work using :

(*a*) the Callow upward-flow classifier,
(*b*) a series of conical glass bulbs (Fig. 4),

it was established that the hydraulic classification of low-grade torbanite depended on three factors :

(i) the velocity of flow of water,
(ii) the free-falling velocities of the particles,
(iii) the size of the particles.

In the tests described, all three factors were variable, although the velocity of water-flow throughout any test was maintained as constant as possible.

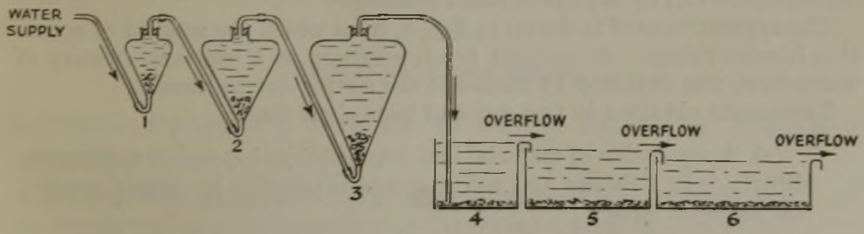

FIG. 4.

CONICAL BULB CLASSIFICATION.

TABLE III.

Hydraulic Classification Tests on Torbanite.

Test No.	Apparatus used.	Size of particles.	Products in order.	Products on original, %.	Dry basis.			
					Vols., %.	Ash, %.	Fixed carbon, %.	Fuel ratio.
Original material (Sample No. 13—Giesecke Adit)					30·00	41·20	28·80	1·04
I	Callow upward-flow classifier	(See grading analysis)	A	15·42	25·99	43·44	30·57	0·85
			B	23·35	25·82	42·07	32·11	0·81
			C	28·64	26·78	41·38	31·84	0·84
			D	28·19	26·20	44·22	29·58	0·89
			Loss	4·40	—	—	—	—
			Total	100·00				
II	Conical glass bulbs	(See grading analysis)	A	61·5	28·94	40·22	30·84	0·94
			B	14·5	30·13	41·74	28·13	1·07
			Loss	24·0	—	—	—	—
			Total	100·0				
III	Conical glass bulbs	(See grading analysis)	A	47·5	25·45	45·90	28·65	0·89
			B *	26·5	30·58	38·74	30·68	1·00
			C †	20·0	32·04	38·57	29·39	1·09
			Loss	6·0	—	—	—	—
			Total	100·0				
IV	Callow upward-flow classifier	(See grading analysis)	A	24·68	27·01	42·62	30·37	0·89
			B	23·35	27·71	39·40	32·89	0·84
			C	18·50	29·43	41·31	29·26	1·01
			D	28·63	28·89	42·85	28·26	1·02
			Loss	4·84	—	—	—	—
			Total	100·00				

* B obtained by mixing products in 2 and 3.
† C obtained by mixing products in 4, 5, and 6.

Further, the results obtained were unsatisfactory from an economic point of view, owing mainly to the fact that all the finest material (which, incidentally, was of no better quality than the coarser material—see section on "Grading of Torbanite by Dry Screening") inevitably settled with the overflow irrespective of specific gravity, so that a true specific gravity separation, as desired, could not be effected at any given velocity for ungraded material of greatly varying size.

It was therefore decided to construct a hydraulic classification apparatus in the form of a long glass tube, 145 cm. long and 1·833 cm. internal diameter, which would facilitate specific gravity separation. Since the free-falling velocities of particles of equal size were proportional to their specific gravities, it was decided to classify material of approximately constant size throughout—*i.e.*, by the use of closer grading.

The apparatus used is shown in Fig. 5, from which the method of operation is self-evident. A constant head, resulting in a constant velocity of water-flow, was obtained by means of the simple device shown.

The results obtained in two series of tests show that :

(*a*) A separation of material into two fractions having an appreciable difference in volatiles and ash contents is possible by closely grading the material thus :

$-60+70$; $-70+80$; $-80+90$; $-90+100$; and possibly $-100+120$ I.M.M. mesh.

(*b*) The percentage fixed carbon throughout remains approximately constant and is inappreciably affected by hydraulic classification.

(*c*) The results obtained for material below 120-mesh seem to indicate that no satisfactory separation is possible with the very fine material, owing most probably to the great cohesive affinity of particles of minute size for one another, irrespective of specific gravity.

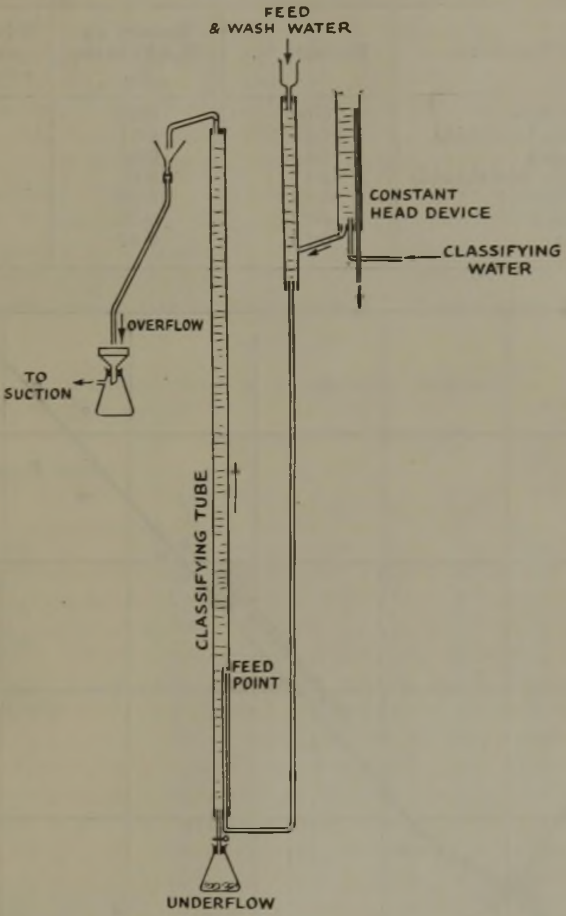

FIG. 5.

LONG TUBE CLASSIFICATION.

(e) *Solvent Extraction of Torbanite by Organic Solvents at Atmospheric Pressures.*

Atmospheric extractions with various organic solvents were carried out by the author on a particularly rich Transvaal deposit of assay value 103·5 gals. per ton (Sample No. 5T) in a Soxhlet apparatus using 50 grams of finely ground material passing a 60-mesh screen. These results are given in

Table IV, showing also the solvent yield as a percentage of the retorting yield. The extract has also been calculated to a dry ash-free basis.

TABLE IV.

Solvent Extraction of Torbanite by Organic Solvents at Atmospheric Pressure.

Sample No. 5T : Retorting yield = 45·1 per cent. oil by weight.

Test No.	Solvent.	Extract, %.	Extract on D.A.F. basis, %.	Solvent yield as percentage of retorting yield.
1	Chloroform	0·60	0·79	1·33
2	Carbon bisulphide	0·54	0·71	1·20
3	Benzene	0·50	0·66	1·11
4	Carbon tetrachloride	0·47	0·62	1·04
5	Shell petrol	0·45	0·59	1·00
6	Acetone	0·42	0·55	0·93
7	Ether	0·36	0·47	0·80

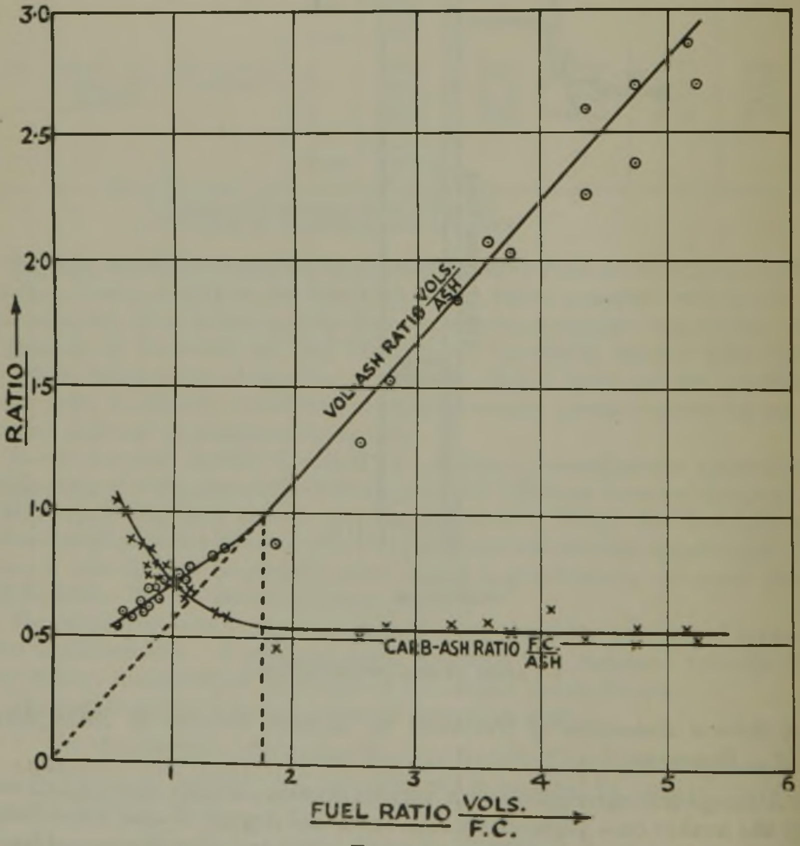

FIG. 6.

CLASSIFICATION OF TORBANITE.

In all cases the extract was either a dark brown oil or semi-solid, giving a yellow to yellowish-brown solution in the solvent.

II. *Chemical Examination.*

(a) *Proximate and Ultimate Analyses.*

In order to obtain results comparable to those of other investigators, at the time of this work the author decided to carry out proximate analyses under the standard conditions for coal—viz., volatiles at 925 ± 25° C., fixed carbon by difference. The author now feels, however, that as torbanite is industrially treated by a low-temperature carbonization process, it is preferable that volatiles should be determined at, say, 550–600° C. In this way, it must be remembered, " fixed carbon " by difference would include combined hydrogen contained in the volatiles (chiefly gaseous) between 575° C. and 925° C., together with that portion of carbon associated with the hydrogen in the volatiles evolved between these temperatures.

Table V gives proximate analyses of a wide range of Transvaal torbanite samples varying in fuel ratio from 5·23 to 0·52. All results have been tabulated on the basis of the dry material, as " moisture " is only adventitious, varying generally from about 0·5 to 2·0 per cent. Values have also been calculated on an ash-free basis in order to investigate the composition of the actual organic matter with which we are more directly concerned.

TABLE V.

Proximate Analyses of Transvaal Torbanite.

No.	Source of sample.	Dry basis.			Dry ash-free basis.		Fuel ratio $=\frac{\text{Vols.}}{\text{F.C.}}$	Vol.–ash ratio $=\frac{\text{Vols.}}{\text{Ash}}$	Carb.–ash ratio $=\frac{\text{F.C.}}{\text{Ash}}$
		Ash, %.	Vols., %.	Fixed C, %.	Vols., %.	Fixed C, %.			
1	Troye Adit	23·68	64·04	12·28	84·1	15·9	5·23	2·71	0·52
2	Carlis' Adit	22·53	64·89	12·58	83·7	16·3	5·16	2·88	0·56
3	" "	23·45	63·25	13·30	82·6	17·4	4·75	2·69	0·57
4	" "	25·64	61·39	12·97	82·5	17·5	4·73	2·39	0·50
5	Troye Adit	23·58	61·40	15·02	80·4	19·6	4·08	2·60	0·64
6	" "	27·24	56·86	15·9	78·2	21·8	3·58	2·09	0·58
7	Carlis' Adit	29·15	54·35	16·50	76·7	23·3	3·30	1·87	0·57
8	" "	32·45	49·56	17·99	73·4	26·6	2·76	1·53	0·56
9	" "	35·9	45·9	18·2	71·8	28·2	2·53	1·28	0·51
10	Troye Adit	42·48	37·47	20·05	65·2	34·8	1·87	0·88	0·47
11	Giesecke Adit	41·9	30·4	27·7	52·5	47·5	1·10	0·73	0·66
12	" "	40·42	30·82	28·76	51·8	48·2	1·07	0·76	0·71
13	" "	41·2	30·0	28·8	51·0	49·0	1·04	0·73	0·70
14	" "	40·78	29·09	30·13	49·1	50·9	0·97	0·71	0·74
15	" "	39·71	28·95	31·34	48·1	51·9	0·93	0·73	0·79
16	" "	42·2	27·3	30·5	47·2	52·8	0·89	0·65	0·73
17	" "	40·19	28·04	31·77	46·9	53·1	0·88	0·70	0·79
18	" "	39·3	27·1	33·6	44·7	55·3	0·81	0·69	0·85
19	" "	42·4	25·7	31·9	44·6	55·4	0·80	0·61	0·75
20	" "	42·4	25·5	32·1	44·3	55·7	0·79	0·60	0·78
21	" "	39·80	25·66	34·54	42·6	57·4	0·74	0·64	0·87
22	" "	40·8	23·4	35·8	39·5	60·5	0·65	0·58	0·89
23*	Artificially deteriorated Giesecke Adit	38·35	23·15	38·5	37·5	62·5	0·60	0·60	1·00
24	Weathered Giesecke Adit	38·35	21·15	40·5	34·3	65·7	0·52	0·55	1·05

* Sample 23 : Kept at 100–150° C. for 1 month.

Fig. 6 has been constructed from the data in Table V, the ratio of volatiles to ash, or, for convenience, vol.–ash ratio, the ratio of fixed carbon to ash, or carb.–ash ratio, being plotted against fuel ratio (*i.e.*, ratio of volatiles to fixed carbon). Table VI gives the ultimate analyses of samples both on a dry and dry ash-free basis.

TABLE VI.

Ultimate Analyses of Transvaal Torbanite.

Sample No.	5T.*	6T.	7C.*	15G.*	17G.
Dry basis:					
C	59·51	58·36			
H	7·25	7·56			
S	1·06 †	1·00	0·90	0·81	0·85
N	0·82	0·76			1·07
O	7·78	5·08			
Dry ash-free basis:					
C	77·87	80·15			
H	9·49	10·40			
S	1·39	1·38	1·27	1·34	1·42
N	1·07	1·05			1·52
O	10·18	6·99			
Ratio:					
C : H	8·21	7·70			
C : O	7·65	11·45			
H : O	0·93	1·49			
Calculated empirical formula of organic material (S + N neglected)	$C_{10\cdot2}H_{14\cdot9}O$	$C_{15\cdot3}H_{23\cdot8}O$			
Calculated composition of total volatiles at 925° C. (*i.e.*, oil + gas + liquor (S + N neglected):					
C	74·75	77·0			
H	12·2	13·75			
O	13·05	9·25			
Empirical formula	$C_{7\cdot6}H_{15}O$	$C_{11}H_{24}O$			
Calculated composition of organic volatiles at 925° C. (*i.e.*, oil + gas) (S + N neglected):					
C	87·6	86·1			
H	12·4	13·9			
Ratio C : H	7·08	6·26			
Empirical formula	$CH_{1\cdot70}$	$CH_{1\cdot93}$			
Calorific value, B.Th.U./lb.:					
Dry basis	13,165	13,110			
Dry ash-free basis	17,230	18,050			

* T. = Troye Adit; C. = Carlis' Adit; G. = Giesecke Adit.
† Consists of 0·94% total organic sulphur and 0·12% inorganic sulphur.

(b) *Experimental Apparatus Used for Carbonization Tests.*

The experimental apparatus used by the author in his carbonization tests, shown diagrammatically in Fig. 7, was devised in accordance with certain basic major principles, generally recognized in carbonization work, and discussed more fully in Section A of this paper. The author has used his discretion in selecting those conditions which were most likely to give optimum results in relation to the particular problem investigated.

A rotary retort was made from 6-inch iron pipe, about 3½ feet in length, with bolted flanges at the ends.

The optimum conditions of retorting were standardized by making the following provisions:

(i) By arranging the retort horizontally so as to spread out the charge, and thus utilize the maximum surface area.

(ii) By uniform external heating obtained from a series of six or

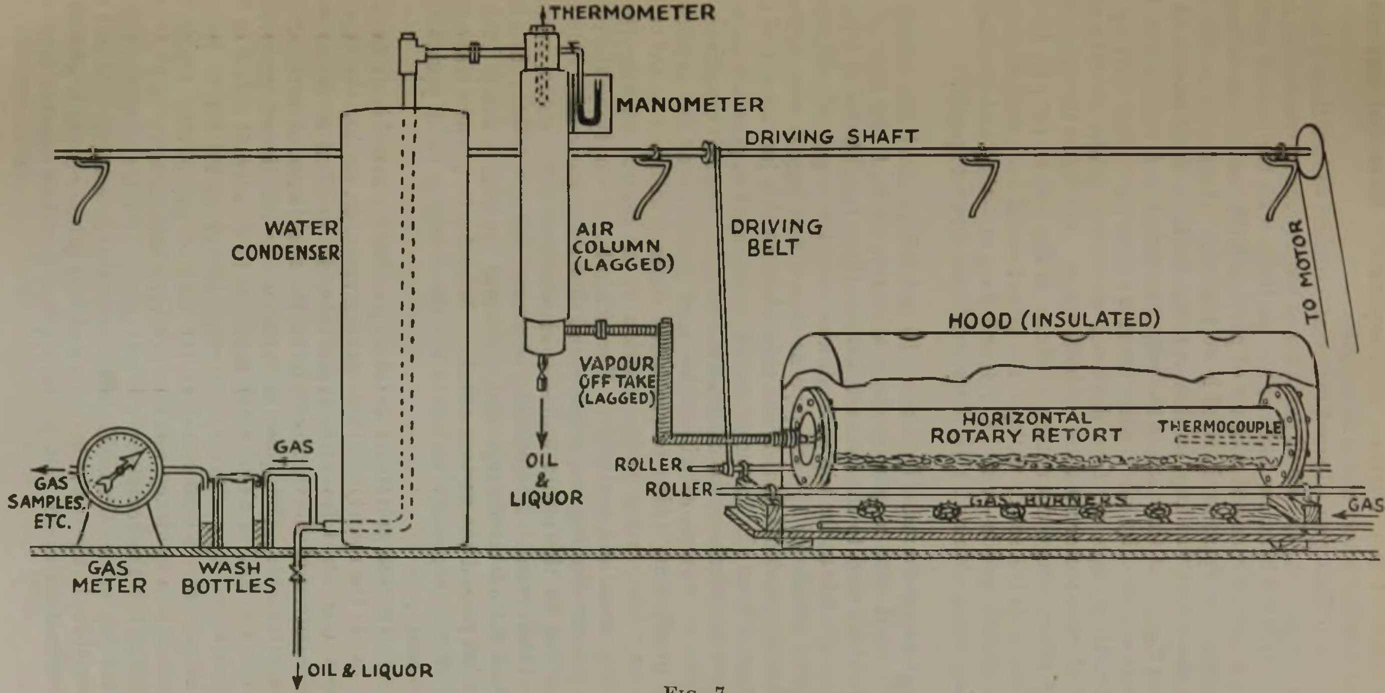

Fig. 7.

EXPERIMENTAL CARBONIZATION APPARATUS.

(CHARGE : 14 LB.)

seven gas-fired ring burners, each separately controlled and placed underneath the retort along its length.

(iii) By utilizing small-sized material, crushed to less than ¼ inch.

(iv) By keeping the small particles in continuous movement so as to secure repeated brief contacts of the particles with the heated surface, giving constant renewed contact of particle with particle, thus largely increasing the opportunities of heat transfer. As shown in the diagram, the retort was kept in slow rotation by the rolling motion of the 11-inch flanges at the ends of the retort on two 1-inch rollers with ball bearings actuated by a belt drive from a ¼-inch steel shaft (157 r.p.m.) fitted with pulleys of three different sizes and driven directly off a ¼-h.p. electric motor rotating at 1460 r.p.m. Owing to the large ratio of the retort flange diameters to those of the rollers (11 : 1), in conjunction with the set of pulleys, the retort could be operated at three different slow speeds—viz., 18, 7·4, or 3·9 r.p.m.—the most satisfactory running being obtained with the second and slowest speeds.

The retort was surrounded by a hood, 18 inches high and hemi-cylindrical at the top, lined with a quarter-sheet asbestos, so that the heat of the burners could be used to the best advantage, three small holes of 3 inches diameter being made at the top through which the burnt gases could escape.

The vapour offtake of 1¼-inch pipe as shown was lagged with asbestos rope, so that no condensation should take place at this juncture, and that vapour removal from the hot zone should be as rapid as possible, in order to keep down the tendency for oil cracking, with consequent enrichment of the gas—both as regards calorific value and quantity—at the expense of the oil, by overheating owing to prolonged contact with hot metal or hot shale. In any case, owing to the rotation of the retort, cracking effects were generally small and negligible. For example, on emptying the retort after each test for cleaning out, it was found that there was only a very thin crust of carbon on the retort walls—evidence that some slight cracking must have taken place. The offtake, of course, did not rotate, the gland being packed with asbestos rope and tightened up with a 2-inch bushing through which passed the vapour offtake. In order to prevent leakage at this point it was necessary to screw up the bushing tight periodically throughout a run.

Dust carry-over was prevented as far as possible by attaching a bend to the offtake inside the retort, with the opening upwards, so that the vapours passed out of the retort from the upper side, the crushed material being mainly at the bottom of the retort, except for the small amount carried round by centrifugal action. The speed of rotation, as mentioned previously, was, however, too low to allow much material to be carried round in this way. Nevertheless, the offtake on several occasions tended to become plugged. A perforated disc fitted at the end of the offtake inside the retort to reduce this tendency was also not very satisfactory.

As a result of this dust carry-over it was necessary after all tests to clean out the offtake as well as the inside of the retort after cooling, and add the dust to the residue in the retort before a material balance could be obtained. A similar correction often had to be applied for dust carried into the condensers.

After removal of the condensable products—viz., oil and liquor—the gas containing light spirit was in several instances scrubbed by passing through a series of two wash-bottles containing high-boiling mineral-gas oil. The dry gas was then metered, as required, and burnt.

As one of the objects of the retorting tests carried out by the author was to examine the effect of temperature on the course of the carbonization, and as the runs were on fixed batches initially at atmospheric temperature, a maximum value of 600° C. was decided on for all tests, so that the efficiency of oil production should be as high as possible.

According to the foregoing discussion on the effects of varying rates of heating, it was decided that medium rates of heating were on the whole advisable, and so all the carbonization runs were carried out to give the maximum temperature of 600° C. within about 5 or 6 hours, with variations from about 3 to 7 hours.

The retort was charged with 14 lb. of the crushed torbanite, and the burners were adjusted to give the desired rate of heating.

The temperature of the retort was taken by a thermocouple placed in a well at the centre line of the retort and about 10–12 inches from the end. The temperature recorded on the instrument was thus that of the torbanite material, and not that of the vapour. It is to be noted that in all these experiments the recorded retort temperatures are those of the central core of the charge, and there was undoubtedly a fairly high temperature differential between the outer wall of the retort and the centre of the core.

A water manometer connected to the top of the air column recorded the pressure at this point during carbonization, steady conditions in the retort being maintained at about 2–3 inches water pressure.

(c) *Carbonization Tests on Transvaal Torbanite.*

The author has carried out carbonization tests with the experimental rotary retort on various grades of torbanite assaying from only about 13 gallons per ton to 106 gallons per ton, with fuel ratios varying from 0·52 to 5·23, respectively. The results of these tests have been summarized in Table VII.

Material balances have been drawn up for each test, so that a direct comparison of different torbanite grades can be made at a glance. Oil and liquor yields have also been calculated to a basis of gallons per short ton (2000 lb.).

The importance of bringing results to a dry ash-free basis has not been overlooked, as, after all, we are concerned only with the quality of the organic matter, and not with that of the mineral matter associated with it.

In order to obtain an idea of the degree of reproducibility obtainable with the experimental apparatus, six tests on the same sample (No. 27G) were carried out, conditions throughout each test being maintained as nearly as possible the same. The results obtained and tabulated in Tests 17a to 17f are reasonably constant for this type of work, so that it may be assumed that all the tests carried out on the other samples under identical conditions are immediately comparable with each other.

Test 18 was carried out with the object of determining the effect of retorting a mixture of samples of different grades, and from a comparison of the

TABLE

Carbonization of Transvaal

(14-lb. Charges in Experi

Test No.:	1.	2.	3.	4.	5.	6.	8.	9.	10.	11.	12.
Sample No. and source: (See Table V.)	1T.*	2C.*	6T.	7C.	8C.	25T.	10T.	14G.*	15G.	17G.	18G.
Material balance:											
Spent shale, %	44·1	41·8	42·2	66·30	53·23	55·8	68·6	77·09	73·9	75·65	75·3
Oil, %	45·3	46·1	44·85	20·41	34·00	31·0	18·7	11·03	12·2	11·89	11·0
Total liquor, %	2·5	3·0	3·15	4·22	4·66	5·4	3·8	4·69	5·1	7·79	7·6
Gas—loss, %	8·1	9·1	9·8	9·07	8·11	7·8	8·9	7·19	8·8	4·67	6·1
Oil:											
Yield, in gals. per ton	106·4	104·5	103·0	49·5	81·5	72·7	43·3	25·3	26·9	26·0	24·1
Yield, in G.P.T. (D.A.F. basis)	140·2	135·5	141·5	70·2	122·1		76·3	43·8	45·8	44·6	40·4
Gravity, at 15·5° C.	† 0·851	0·833	0·872	0·865	0·866	† 0·852	† 0·870	0·872	0·908	0·919	0·915
Tar acids in oil, %		2·2	1·98			4·0			4·95	2·85	2·1
Tar bases in oil, %		0·9	1·01			0·8			1·14	2·42	
Liquor:											
Total yield, gals. per ton	5·0	6·0	6·3	8·4	9·3	10·8	7·6	9·4	10·2	15·6	15·2
Liq. of composition, G.P.T.	3·9	5·1	6·3	6·2	7·1		5·1	4·5	5·4	10·7	11·5
Liq. of decomposition, G.P.T. (D.A.F. basis)	5·1	6·6	8·6	8·8	10·6		9·0	9·8	9·2	18·3	19·3
Total $(NH_4)_2SO_4$, % in liquor		3·4							1·7	5·3	
Free $NH_3AS(NH_4)_2SO_4$, %		2·5							1·2	3·4	
Total $(NH_4)_2SO_4$, lb. per ton		2·0							1·7	8·3	
Vol. ratio: Yield oil/Yield liquor of decomposition	27·5	20·6	16·4	8·0	11·5		8·5	5·6	5·0	2·4	2·1
Gas:											
Yield, cu. ft. per ton at N.T.P.	2300		1880	3140	2640	1940	2135	2060	2010	2285	1890
Yield, cu. ft./ton at N.T.P. (D.A.F. Basis)	3030		2585	4450	3955		3760	3560	3410	3920	3170
Weight ratio: oil/gas	5·6	5·1	4·6	2·3	4·2	4·0	2·1	1·5	1·4	2·5	1·8
Proximate analyses:											
Raw material—											
Moisture, %	0·53	0·45		1·12	1·09		1·24	2·45	2·39	2·45	1·86
Volatiles, %	63·71	64·61	56·86	53·74	49·01		37·01	28·38	28·25	27·35	26·60
Ash, %	23·55	22·42	27·24	28·82	32·10		41·96	39·77	38·76	39·20	38·52
Fixed carbon, %	12·21	12·52	15·90	16·32	17·80		19·79	29·40	30·60	31·00	33·02
Fuel ratio	5·23	5·16	3·58	3·30	2·76		1·87	0·97	0·93	0·88	0·81
Vol.–ash ratio	2·71	2·88	2·09	1·87	1·53		0·88	0·71	0·73	0·70	0·69
Carb.–ash ratio	0·52	0·56	0·58	0·57	0·56		0·47	0·74	0·79	0·79	0·85
Spent shale—											
Volatiles, %		5·36	2·9	17·57	4·51			5·28	2·57	3·39	
Ash, %		55·50	56·7	47·44	57·38			52·47	54·08	51·89	
Fixed carbon, %		39·14	40·4	34·99	38·11			42·25	43·35	44·72	
Fuel ratio		0·14	0·07	0·50	0·12			0·13	0·06	0·08	
Vol.–ash ratio		0·10	0·05	0·37	0·08			0·10	0·05	0·07	
Carb.–ash ratio		0·71	0·72	0·74	0·66			0·81	0·80	0·86	
Spent shale reduced to original material—											
Volatiles, %		2·16	1·39	10·69	2·52			4·00	1·84	2·56	
Ash, %		22·42	27·24	28·82	32·10			39·77	38·76	39·20	
Fixed carbon, %		15·82	19·4	21·28	21·31			32·01	31·05	33·81	
		40·40	48·03	60·79	55·93			75·78	71·65	75·57	
Decrease in original vols., %		96·5	97·7	80·0	94·8			85·6	93·3	90·6	
Increase in fixed carbon, %		26·3	22·0	30·0	16·6			8·9	1·5	9·0	

* T. = Troye Adit; C. = Carlis' Adit; G. = Giesecke Adit.

VII.

Torbanite at 600° *C.*

mental Rotary Retort.)

13.	14.	15.	16.	17a.	17b.	17c.	17d.	17e.	17f.	17 mean	18a.	18b.	19a.	19b.
26G.	21G.	22G.	24 WEA. G.	27G.	27G.	27G.	27G.	27G.	27G.	27G.	50% of 10T. + 50% of 21G.	Calc. for 50% of 10T. + 50% of 21G.	50% of 1T. + 50% of 21G.	Calc. for 50% of 1T. + 50% of 21G.
78·1	76·5	76·4	78·2	78·0	78·6	78·0	77·5	77·7	77·8	78·0	72·1	72·6	60·8	60·3
9·5	8·8	8·9	7·45	6·5	6·0	5·9	6·6	5·8	5·7	6·1	14·5	13·7	26·3	27·0
6·5	6·3	8·0	7·55	6·5	7·0	7·0	6·8	7·3	6·8	6·9	4·8	5·0	4·7	4·5
5·9	8·4	6·7	6·8	9·0	8·4	9·1	9·1	9·2	9·7	9·0	8·6	8·7	8·2	8·2
21·2	19·6	19·7	16·45	14·5	13·4	12·9	14·5	13·0	12·4	13·5	32·8	31·4	61·6	63·3
	33·4	34·0	27·2											
0·897	† 0·899	0·901	0·904	0·910	0·902	0·915	0·917	0·902	0·920	0·911	† 0·895	† 0·880	† 0·860†	† 0·857
5·9		4·5	4·1	5·9	5·9	5·7	6·6	6·3	7·1	6·3				
0·74		1·6	3·8											
13·0	12·6	16·0	15·1	13·0	14·0	14·0	13·6	14·6	13·6	13·8	9·6	10·0	9·4	9·0
	8·1	11·7	10·7											
	13·8	19·8	17·7											
			3·5											
			5·0											
	2·4	1·7	1·5											
1590	2525	1135	1345	2415	2495	2330	2550	2555	2580	2485	2235	2330	2465	2420
	4300	1960	2225											
1·6	1·1	1·3	1·1											
	2·27	2·16	2·18											
	25·09	22·87	20·72											
	38·90	39·82	37·46											
	33·74	35·15	39·64											
	0·74	0·65	0·52											
	0·64	0·58	0·55											
	0·87	0·89	1·05											
		3·66												
		50·62												
		45·72												
		0·08												
		0·07												
		0·90												
		2·88												
		39·82												
		36·00												
		78·70												
		87·0												
		2·4												

† Oil includes scrubbed spirit from gas (up to 2 gals. per ton raw torbanite).

calculated values the conclusion was reached that a mixture of different qualities of oil shale yields distillation results which are the average of the samples retorted, each apparently behaving independently of the other.

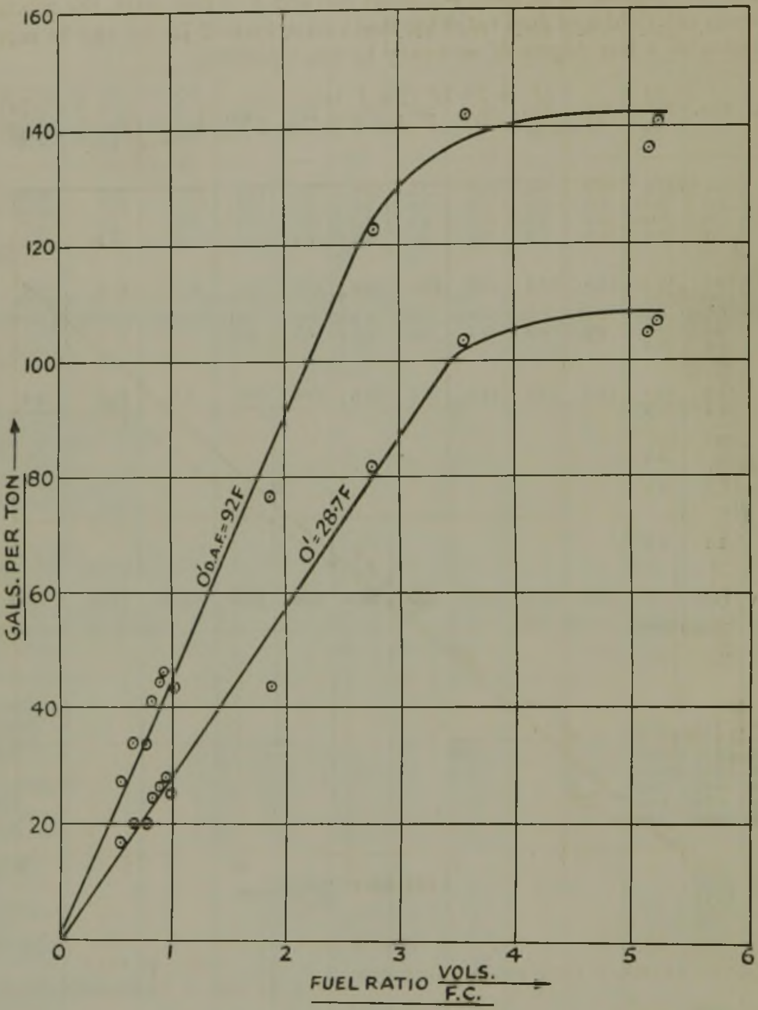

FIG. 8.

RELATION OF OIL YIELD TO FUEL RATIO.

Test No. 19 was carried out on two samples showing a greater difference in quality than those used in Test 18, similar conclusions being reached.

Fig. 8 has been drawn to show the relation between oil yield and fuel ratio. This shows that for fuel ratios up to about 2·75–3·5 the oil yield increases steadily with fuel ratio and is approximately directly proportional to it. For material of higher fuel ratio, a large increase of fuel ratio is accompanied by only a comparatively small increase in oil content. Curves have been plotted for the actual material as mined and also on a dry ash-free basis.

If O' = yield of oil in gals. per short ton and F = fuel ratio, the relation between oil yield and fuel ratio for fuel ratios up to 2·75–3·5 can be represented with a fair degree of accuracy by the equations :

$$O' = 28{\cdot}7F \text{ (for } F \text{ below } 3{\cdot}5) \quad . \quad . \quad . \quad . \quad . \quad (a)$$

and

$$O'_{\text{D.A.F.}} = 92 \ F \text{ (for } F \text{ below } 2{\cdot}75) \quad . \quad . \quad . \quad . \quad . \quad (b)$$

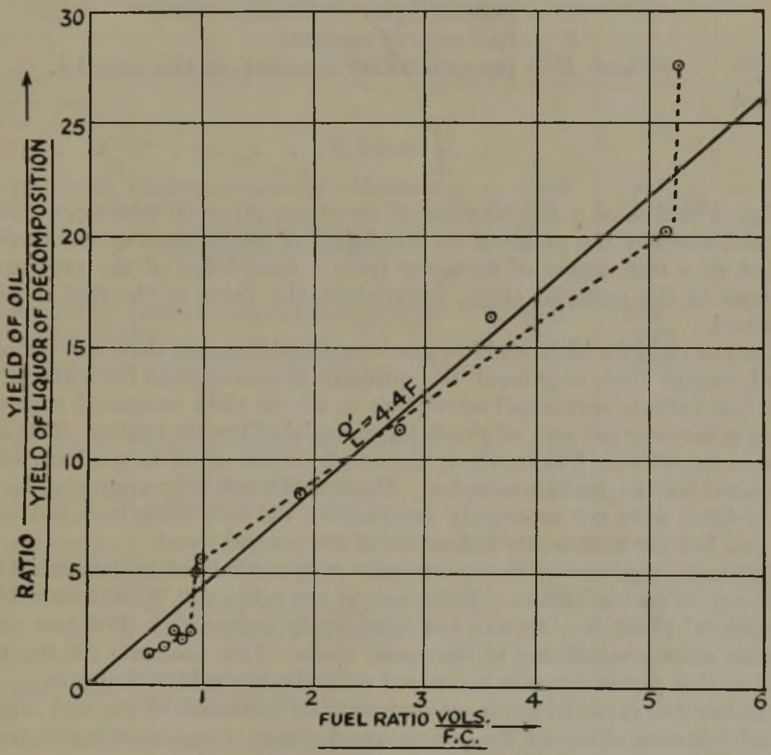

FIG. 9.

RELATION BETWEEN FUEL RATIO AND RATIO $\frac{\text{YIELD OF OIL}}{\text{YIELD OF LIQUOR OF DECOMPOSITION}}$.

These curves or equations might be usefully applied for predicting approximate oil yields of different samples of torbanite from a knowledge of the fuel ratios alone.

As far as the liquor is concerned, other authors apparently do not differentiate between incidental moisture present in the shale and liquor formed from actual decomposition of the organic matter. The present author is of the opinion that such a distinction is essential.

The liquor yield decreases with increase of fuel ratio, so that the ratio of yield of oil to that of liquor of decomposition increases rapidly with increase of fuel ratio. This relation has been represented graphically in dotted lines in Fig. 9, but from the data available it appears that for practical purposes the relation between the ratio of yield of oil to that of liquor of decomposition and the fuel ratio of the material can be approximately represented by a straight-line graph of general equation

$$\frac{O'}{L} = KF$$

where O' = yield of oil,
L = yield of liquor of decomposition,
F = fuel ratio of material,
and K = proportionality constant, in this case 4·4.

so that

$$\frac{O'}{L} = 4{\cdot}4\,F \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (c)$$

Figs. 8 and 9, or a combination of equations (*a*) or (*b*) with (*c*), afford a method whereby the yields of oil and liquor of decomposition can be estimated to a fair degree of accuracy from a knowledge of the proximate analysis of the material alone, from which the value of the fuel ratio is obtained.

The gas yield for high- and low-grade material does not show any definite trend, except that, in general, the quantity of gas evolved for material of high fuel ratio is very small in relation to the oil yield compared with the relation between gas and oil yields for material of low fuel ratio. This can be seen from Table VII in which the weight ratios of oil to gas have been tabulated for the various samples. These ratios are only approximate, as gas weights were not separately determined, the loss being included with the gas, but are sufficiently indicative of the general trend.

Proximate analyses of the spent shale were carried out to determine the efficiency of carbonization. Reference to the table will demonstrate that Sample 7C (Test No. 4) was not completely carbonized, 17·6 per cent. volatile matter remaining in the spent shale. This accounts for the low yield in this particular case compared with the fairly high fuel ratio.

The low fuel ratios of the spent shales are an indication of the high degree of carbonization obtained, the percentage decrease in organic volatiles being regarded as the actual efficiency of retorting.

Table VIII gives an idea of the distribution of sulphur in the products obtained from low-temperature carbonization of torbanite.

From the determinations made the author has calculated also the percentage of " volatile " and " fixed " sulphur, as it is these sulphur figures which are of paramount importance when considering the potentialities of the shale as a raw material for the production of a low-sulphur-bearing crude oil.

The sulphur in both raw and spent shale was determined in the well-known method of igniting with Eschka's Mixture and estimating sulphates by precipitation with barium chloride.

The sulphur in the gas was assumed to consist of H_2S only. For sample No. 15G, the hydrogen sulphide was absorbed in the towers packed with porcelain beads, the gas being passed in counter-current to a slow stream of lead acetate solution. The precipitated sulphide was washed, dissolved in nitric acid, and the lead determined as chromate after the addition of sodium acetate.

For Sample No. 7T, the gas was purified from hydrogen sulphide by means of a commercial iron oxide purifier as used in coal-gas purification, the gas being sucked through by a suction pump. The sulphur in the gas was then obtained by analysis of the purifier before and after gas purification.

TABLE VIII.

Sulphur Distribution in Torbanite Low-Temperature Carbonization Products.

Sample No.:	7T.	15G.	17G.
Raw torbanite:			
Per cent. total sulphur	0·90	0·81	0·85
Gas:			
Per cent. by weight on original raw torbanite .	9·07	6·5	
Per cent. sulphur	4·7	2·43	
Per cent. ratio $\frac{\text{sulphur in gas}}{\text{sulphur in raw torbanite}}$ . .	47·3	17·7	
Oil:			
Per cent. by weight on original raw torbanite .	20·41	15·2	
Per cent. sulphur	0·21	0·65	
Per cent. ratio $\frac{\text{sulphur in oil}}{\text{sulphur in raw torbanite}}$ . .	4·8	12·3	
Liquor:			
Per cent. by weight on original raw torbanite .	4·22	5·4	
Per cent. sulphur	0·02	0·08	
Per cent. ratio $\frac{\text{Sulphur in liquor}}{\text{sulphur in raw torbanite}}$ . .	0·1	0·5	
Spent torbanite:			
Per cent. by weight on original raw torbanite .	66·3	72·9	76·3
Per cent. sulphur	0·63	0·75	0·59
Per cent. ratio $\frac{\text{Sulphur in spent torbanite}}{\text{sulphur in raw torbanite}}$.	46·0	67·5	52·9
Per cent. ratio $\frac{\text{sulphur unaccounted for}}{\text{sulphur in raw torbanite}}$ . .	1·8	1·2	
Per cent. total "volatile" sulphur (*i.e.*, in oil, gas and liquor) (based on original raw torbanite). .	0·49	0·26	0·40
Per cent. "fixed" sulphur (*i.e.*, in spent torbanite) (based on original raw torbanite) . . .	0·41	0·55	0·45
Per cent. ratio $\frac{\text{total ``volatile'' sulphur}}{\text{``fixed'' sulphur}}$ . .	1·20	0·47	0·89

The sulphur in the crude water-free oil was estimated by the "Lamp Method," using as solvent amyl acetate, the procedure described by Esling[18] being followed, the sulphur dioxide being absorbed by a caustic soda solution followed by a guard tower of ammonium carbonate and estimated gravimetrically as sulphate.

The sulphur in the crude water liquor separated from the oil, after oxidation with nitric acid, was also determined gravimetrically as sulphate.

Determinations on the nitrogen content of Sample No. 17G and its residue after carbonization have shown that:

Nitrogen in raw torbanite	= 1·07 per cent.
Nitrogen in spent torbanite	= 0·97 ,, ,,
Giving total " volatile " nitrogen (*i.e.*, in oil, gas and liquor)	= 0·33 ,, ,,
And " fixed " nitrogen (*i.e.*, in spent torbanite)	= 0·74 ,, ,,

corresponding to 0·40 per cent. ammonia recovery (based on original torbanite) or 1·56 per cent. as ammonium sulphate, or about 30 lb. per short ton—*i.e.*, 69·2 per cent. of the original nitrogen content on simple low-temperature carbonization is irrecoverable as ammonia.

A determination of total ammonium sulphate in the liquor obtained by carbonization gave 8·3 lb. per short ton.

(d) *A Study of the Progress of Carbonization.*

During the carbonization tests carried out by the author with the experimental rotary retort, at regular time intervals of 15 minutes, readings of time, shale temperature, oil, liquor, and gas volumes were taken.

From these results the following are the principal observations:—

1. Rich torbanite begins to decompose at 300° C., maximum decomposition setting in between 350° and 425° C., about 90–95 per cent. of the oil being evolved up to 450° C., very little additional oil being evolved above 500° C.
2. Poorer torbanite begins to decompose, however, at about 350° C. with steady decomposition up to 550° C. when 95 per cent. of the oil has been evolved, 85–90 per cent. being evolved at 500° C. From 550° to 600° C. the final 5 per cent. of oil is obtained.
3. Liquor from both high- and low-grade torbanite begins to distil at about 150° C. retort temperature, the liquor at this stage being chiefly the incidental. Up to about 350° C. for high-grade material very little actual liquor of decomposition is obtained, the corresponding temperature for lower-grade material being somewhat lower—about 300° C. From these points liquor of decomposition is evolved at a fairly steady rate to 600° C.
4. Very little gas is evolved up to 350° C. for both classes of torbanite, from which point gas is evolved steadily to 600° C., with the difference that for high-grade material the tendency is for more rapid gas evolution up to about 475° C., evolution becoming somewhat slower above this temperature when 65–75 per cent. of the total gas to 600° C. has been obtained, whereas for the lower-grade mineral only about 40–50 per cent. of the total gas to 600° C. is evolved up to 475° C.

(e) *Effect of Temperature of Evolution on Oil Composition.*

In order to obtain an idea whether there is any considerable difference in the gravities and distillation ranges of the oils distilling off from torbanite at different temperatures, during one test (No. 3) on Sample No. 6T it was decided to examine separately the fractions evolved up to 350° C., 350–400° C., 400–450° C., 450–500° C., 500–600° C., and also the spirit obtained by scrubbing the gas.

The results obtained in these determinations are given in Table IX.

TABLE IX.

Effect of Temperature of Evolution on Instantaneous Composition of Oil.

Test No. 3. Sample No. 9T.

Retort temp. :	To 350° C.	350–400° C.	400–450° C.	450–500° C.	500–600° C.	Scrubbed spirit.	Total average oil.
Oil vol., %	3·7	36·8	52·8	3·9	1·95	0·85	100·0
Oil gravity at 15·5° C.	0·853	0·863	0·873	0·884	0·896	0·800	0·872
Oil distillation, % :							
To 100° C.	1·3	2·2	2·8	5·9	14·1	23·6	3·2
,, 125	4·4	4·0	6·3	9·8	20·3	43·7	6·8
,, 150	7·6	6·0	10·8	14·2	24·4	91·0	10·0
,, 175	12·0	8·6	14·7	18·0	30·0	100·0	13·1
,, 200	17·0	12·4	16·6	21·4	36·4		17·0
,, 225	22·8	19·4	20·3	24·4	40·6		21·2
,, 250	30·9	23·8	24·9	27·4	46·7		25·7
,, 275	41·0	30·0	29·8	30·8	50·1		30·8
,, 300	53·0	38·3	37·8	36·6	53·1		38·1
,, 325	64·5	53·3	55·4	43·4	57·2		53·1
,, 350	76·4	60·4	72·1	51·7	63·5		64·6
,, 360		64·6	81·5	62·0	67·8		71·5
Residue, etc.	23·6	35·4	18·5	38·0	32·2		28·5

(f) *Composition of the Oil.*

The distillation ranges of the various oils obtained in the carbonization tests described are shown in Table X, together with the results obtained by distillation of an average sample of Giesecke oil made on a large scale in the Salermo retorts at Ermelo.

Although there are apparent differences in the relative proportions of low- and high-boiling constituents, judged by the distillation analysis, the conclusion is that the oils are more or less of equal quality, differing more in degree than in character.

From a specific-gravity study, however, the wider fluctuations point to differences in quality, the tendency being marked for the better class of torbanite from the Carlis' and Troye Adits to produce an oil of lower gravity and generally of higher quality than the poorer Giesecke grade. The density on the whole is higher than that of petroleum oil, but not as high as that of coal tar.

The crude torbanite oil from retorting is a dark-green liquid with a disagreeable odour smelling slightly of H_2S. The average sulphur value of Transvaal torbanite oil obtained by the author for a large number of samples is about 0·45 per cent., varying from 0·21 to 0·65 per cent. The oil is highly unsaturated, as evidenced by its solubility in sulphuric acid.

TABLE X.

Distillation Ranges of Oil from Transvaal Torbanite.

Test No.:	2.	3.	4.	6.	8.	9.	10.	11.	12.	13.	14.	15.	16.	Average Giesecke oil on large scale.
Sample No. and source.	2C.*	6T.*	7C.	25T.	10T.	14G.*	15G.	17G.	18G.	26G.	21G.	22G.	24 W.G.	
Specific gravity at 15·5° C.	0·857	0·872	0·865	†0·852	†0·870	0·872	0·908	0·919	0·915	0·897	†0·899	0·901	0·904	0·900
To 100° C., %	1·6	3·2	1·2	2·1	2·4	1·3	1·4	0·7	0·6	1·7	3·5	3·1	0·7	2·8
,, 125	3·0	6·8	2·8	4·1	4·0	4·8	3·2	1·9	2·6	3·7	6·4	6·1	1·8	6·0
,, 150	5·8	10·0	7·7	9·1	9·0	6·9	5·2	4·0	5·5	8·0	9·6	11·2	4·8	11·0
,, 175	9·0	13·1	12·9	15·0	15·0	11·0	9·6	7·6	11·4	11·0	14·8	13·8	10·7	15·5
,, 200	16·4	17·0	18·7	22·3	22·0	17·5	15·3	16·3	19·0	16·0	19·8	17·2	16·9	19·5
,, 225	21·6	21·2	23·5	27·7	27·0	24·8	21·4	21·0	26·2	22·5	26·2	22·2	23·3	23·2
,, 250	27·0	25·7	28·0	33·6	32·5	32·9	34·3	28·6	35·6	31·6	31·5	27·5	31·2	30·8
,, 275	36·3	30·8	34·0	39·2	39·0	42·3	39·4	38·0	41·7	38·0	35·0	32·6	39·6	36·5
,, 300	46·9	38·1	43·6	47·6	47·5	50·8	51·5	47·0	52·7	50·0	40·0	47·8	48·2	44·0
,, 325	51·0	53·1	50·9	53·6	54·0	60·5	64·3	57·2	61·2	63·3	49·1	47·2	58·4	55·1
,, 350	65·2	64·6	63·4	61·7	62·3	70·8	71·0	70·0	72·6	70·3	61·0	60·2	67·4	68·2
,, 360	73·9	71·5	77·6	65·0	66·2	76·4	78·6	75·7	76·9	77·0	70·1	67·3	75·0	75·0
Residue, etc.	26·1	28·5	22·4	35·0	33·8	23·6	21·4	24·3	23·1	23·0	29·9	32·7	25·0	25·0

* C = Carlis' Adit; T = Troye Adit; G = Giesecke Adit.
† Includes scrubbed spirit.

TABLE XI.

Actual Gas Analyses.

(Intermediate values.)

Gas sample:	A.	B.	C.	D.	E.	F.	Representative sample (including samples A to F).
Temp. range	300–350° C.	350–400° C.	400–450° C.	450–525° C.	525–600° C.	At 600° C.	(300–600° C.)
H_2S	5·0	4·3	3·5	2·1	0·4	0·5	1·7
CO_2	15·1	5·7	8·6	6·5	1·8	4·3	5·8
C_nH_m	12·3	11·0	1·3	0·3	Nil	Nil	2·6
O_2	1·2	1·0	0·8	0·5	0·8	0·8	1·0
CO	5·6	4·6	8·6	10·9	12·0	14·1	10·2
H_2		27·6	47·3	56·7	64·5	59·9	40·2
C_2H_6	31·9						
Saturates as CH_4	23·2	33·7	19·0	14·0	9·3	9·7	26·6
N_2	5·7	12·1	10·9	9·0	11·2	10·7	11·9
Wt. of gas at N.T.P., gms./litre	1·279	0·805	0·673	0·571	0·468	0·537	0·690
Weight of gas at 20° C. and 642 mm. of Hg, gms./litre	1·003	0·633	0·530	0·449	0·368	0·423	0·542
Specific gravity of gas (air = 1)	0·989	0·621	0·520	0·442	0·362	0·416	0·533
Gross calorific value at N.T.P., B.Th.U./cu. ft.	1122	682	439	400	364	360	511
Nett calorific value at N.T.P., B.Th.U./cu. ft.	1008	619	391	354	320	318	458
Gross calorific value at 30 ins. 60° F. saturated, B.Th.U./cu. ft.	1048	636	410	373	340	336	476
Nett calorific value at 30 ins. 60° F. saturated, B.Th.U./cu. ft.	942	577	364	330	298	296	427

TABLE XII.

Calculated Gas Analyses.

(Cumulative values.)

Gas sample:	300–350° C.	Total gas to 400° C.	Total gas to 450° C.	Total gas to 525° C.	Total gas to 600° C.	Calculated representative sample.	Corrected actual representative sample.
H_2S	5·0	4·7	4·1	3·4	2·6	2·3	1·7
CO_2	15·1	10·7	9·7	8·5	6·8	6·3	5·8
C_nH_m	12·3	11·7	6·9	4·5	3·3	2·8	2·6
O_2	1·2	1·0	0·9	0·8	0·8	0·8	1·0
CO	5·6	5·1	6·7	8·3	9·3	9·9	10·2
H_2		13·1	29·0	39·1	45·9	47·5	46·7
C_2H_6	31·9	45·0	33·0	26·0	21·4	20·4	6·5
Saturates as CH_4	23·2						13·6
N_2	5·7	8·7	9·7	9·4	9·9	10·0	11·9
Wt. of gas at N.T.P., gms./litre	1·279	0·949	0·821	0·729	0·660	0·643	0·689
Weight of gas at 20° C. and 642 mm. of Hg, gms./litre	1·003	0·747	0·645	0·574	0·520	0·506	0·542
Specific gravity of gas (air = 1)	0·989	0·733	0·634	0·563	0·511	0·497	0·533
Gross calorific value at N.T.P., B.Th.U./cu. ft.	1122	770	618	538	490	477	516
Nett calorific value at N.T.P., B.Th.U./cu. ft.	1008	718	576	503	457	445	482
Gross calorific value at 30 ins. 60° F. saturated, B.Th.U./cu. ft.	1048	702	553	483	438	426	462
Nett calorific value at 30 ins. 60° F. saturated, B.Th.U./cu. ft.	942	655	516	376	408	398	431

(g) *Effect of Temperature of Evolution on Composition, Density, and Calorific Value of the Gaseous Products of Carbonization.*

During carbonization test 17e (Table VII) on Sample No. 27G, a representative sample (from 300 to 600° C. and at 600° C.) was taken by collecting every few minutes a definite volume of gas over saline water in a gas aspirator exactly corresponding to the rate of gas evolution during that time interval. The very small volume evolved up to 300° C. was neglected, as in any case it was rich in the air expelled from the apparatus.

The values obtained by analysis with a modified Orsat were used in the calculation of gas densities and also gross and net calorific values. In the estimation of the latter, it has been assumed that the unsaturated C_nH_{2n} are essentially C_2H_4.

From the figures given in Table XI, the values in Table XII were obtained by calculation using the relative proportions of gases collected between any given temperature range as indicated by the particular readings for the carbonization test under consideration.

(h) *The Ash from Spent Torbanite.*

Proximate analyses of the spent torbanite after carbonization are given in Table VII.

Composite samples of ash were prepared by igniting to constant weight the spent shales obtained from the author's carbonization tests. The ashes from the three adits, Giesecke, Troye, and Carlis, were examined, the analyses being shown in Table XIII.

TABLE XIII.

Composition of Ash from Transvaal Torbanite.

Constituent.	Giesecke Adit.	Troye Adit.	Carlis' Adit.
SiO_2, %	63·97	63·11	58·70
Fe_2O_3, %	2·46	34·71	3·50
Al_2O_3, %	31·65		36·25
TiO_2 %	0·70		0·75
CaO %	0·45	0·81	1·18
MgO %	0·65	1·08	0·32
	99·88	99·71	100·70

It is of interest to note that, irrespective of the source and quality of the original mineral, the ash composition has been found to be reasonably uniform.

In all three cases the ash was of a very good colour—creamy-white. The fusion points were also determined by comparison with standard Seger cones, and for all three samples were found to be above 1500° C.

(j) *Cracking Test of Transvaal Torbanite Crude Oil.*

The technique employed by the author in cracking tests was briefly to remove light spirit by straight distillation, or topping the crude oil and subjecting the topped crude to cracking conditions, forming cracked petrol, coke or bituminous residue, and intermediate heavy oil. A heavy second crack on the recycle gas oil was also carried out, as results more closely approximating to industrial practice could be obtained in this way. Any uncracked oil released in the earlier stages of the test to maintain constant pressure conditions could thus undergo cracking during the second run.

A Hempel distillation on 750 mls. of the crude oil used for the cracking test is given in Table XIV, efficient fractionation being obtained by means of a column of glass beads in the neck of the flask.

TABLE XIV.

Hempel Distillation Characteristics of Torbanite Oil Used for Cracking Tests.

(Gravity 0·885. Hempel distillation (on 750 mls.).)

Distillation, %.	Temperature, ° C.	Fraction and range.	Vol., %.	Gravity at 15·5° C.	Weight, %.
I.B.P.	46	Petrol (to 200° C.) fraction	20·2	0·787	17·5
5	118				
10	148				
15	173				
20	196				
20·2	200				
25	220	Kerosine (200–260° C.) fraction	14·3	0·830	13·5
30	238				
34·5	260				
35	264	Gas oil (260–360° C.) fraction	53·8	0·905	55·0
40	287				
45	301				
50	304				
55	317				
60	339				
65	342				
70	346				
75	351				
80	354				
88·3	360				
Residue: asphaltic and paraffin base				0·996	14·0

About 10 gallons of torbanite oil were topped in a large wood-fired 20-gallon M.S. laboratory still containing a fractionating column made of 3-foot pipe, 5 feet 3 inches high, packed with small stones, lagged with asbestos and connected to a 1¼-inch coiled condenser and light-oil receiver.

A high-pressure bomb was then filled with 3750 mls. (at 25° C.) of the topped crude thus obtained, the conical seating lubricated, and the bomb tightened up. It was then inserted in the previously heated electric furnace, which was maintained at maximum heat throughout.

The pressure started rising rapidly at about 160° C., 2¼ hours after inserting the bomb into the hot furnace. From this stage to the end of the experiment the pressure condition was maintained at about 1000 lb. per square inch average, it being necessary to release the pressure frequently in order to maintain constant conditions varying from 950 to 1050 lb. per square inch.

TABLE XV.

Summary of Results on Cracking Transvaal Torbanite Crude Oil (based on Original Crude).

	Mls.	Vol., %.	Grav.	Gms.	Wts., %.
Torbanite crude oil	4668	100·0	0·885	4130	100·0
1. Petrol:					
(*a*) Topped	943	20·2	0·770	726	17·6
(*b*) First crack	571	12·3	0·767	438	10·6
(*c*) Second crack	605	12·9	0·773	468	11·3
Total petrol	2119	45·4	0·770	1632	39·5
2. Residue:					
(*a*) First crack				377	9·1
(*b*) Second crack				481	11·6
Total residue				858	20·7
3. Gas, etc.:					
(*a*) First crack				432	10·5
(*b*) Second crack				586	14·2
Total gas, etc.				1018	24·7
4. Recycle gas oil (above 200° C.)	681	14·6	0·933	635	15·4
Total	2800	60·0		4143	100·3

The experimental procedure adopted by the author in the cracking test differs from a typical cracking plant in the following respect. In modern cracking practice the residue oil of distillation range higher than that of the gas oil does not usually enter the cracking zone, owing to the danger of excessive cracking. In the author's experiments it was decided to subject to cracking conditions all the oil boiling above 200° C., as there were no tubes which might be coked up in this case, coke, if formed, remaining in the autoclave. In this way the heavy gas oil which would otherwise be previously removed with the residue oil on straight distillation would also be subjected to cracking.

As there was no stirring or shaking mechanism in the autoclave, a certain amount of excessive cracking, with consequent gas formation, and therefore loss of petrol yield, must have taken place owing to the overheating of the bomb. In practice, where such overheating can be avoided and the oil is in motion, the gas formation would not be so high, and therefore the light-oil yield would be higher.

Also, owing to the fact that the light ends, once formed, were not removed from the hot zone fast enough, the experimental conditions were again somewhat more strenuous than in practice, the lighter products being further decomposed to yield gas. This points to the advisability of rapid removal of light-oil vapours—*e.g.*, by a dephlegmator—before excessive cracking can take place.

Another factor worth noting is that the experimental run was a batch process, whereas commercial units are continuous in operation.

From these considerations, one would expect a laboratory cracking bomb-test to give lower petrol yields than would actually be obtained in practice. The total yield in the author's test for topping and two cracks was 45·4 per cent. with a recycle gas oil of 14·6 per cent. A third crack of this recycle oil would undoubtedly give a total yield of about 50 per cent. on the original crude oil, especially as in practice dephlegmation of the light vapours during cracking and condensation under pressure with further recovery from a gas absorption plant all tend to increase the total petrol yield.

In a more recent test on torbanite oil cracking, a procedure was adopted which more closely approximated plant practice, with dephlegmation of vapours in this case and condensation carried out under pressure.

Exhaustive cracking was achieved in this test, the final uncracked recycle oil being only 3·0 per cent. kerosine and 0·8 per cent. gas oil, with a total petrol production of 51·1 per cent.

In this connection it should be noted that yields of 60 per cent. are obtained in actual industrial practice at the Satmar Refinery by combining topping and cracking of Ermelo torbanite oil, in conjunction with the recovery spirit from the wet cracked gas.

SECTION C. DISCUSSION.

The curves in Fig. 6 show that the material torbanite consists of mineral matter which is essentially inherent with the organic constituents which make up the volatile and fixed carbon. Adventitious mineral matter to any appreciable extent is not a characteristic of the material. The regularity of the curves has led the author to the striking conclusion that:

The different grades of Transvaal torbanite, ranging from material yielding a high proportion of oil to inferior material yielding a comparatively small amount of volatiles, may be considered to have a common origin in one parent substance of high fuel ratio. The organic portion of this mineral from various causes has undergone decomposition or "deterioration," leaving the mineral matter to all intents and purposes untouched. First, that part constituting the volatiles has progressively lost material (*e.g.*, by oxidation or by gasification due to release of pressure) until a fuel ratio for the whole mineral of 1·75 has been reached, the fixed carbon content remaining unchanged, as shown by the constancy of the ordinate for the carb.–ash ratio against fuel ratio. That portion of the vol.–ash ratio–fuel ratio curve beyond 1·75 fuel ratio, if projected, passes through the origin, as is to be expected if fixed carbon remains constant. At a fuel ratio of 1·75 the mineral undergoing deterioration becomes subjected to a different change, volatile matter becoming converted into fixed carbon, and it is from this stage that the hydrogen portion of the volatiles is probably attacked to a greater degree than the carbon content (*e.g.*, by oxidation to water), with the resultant effect of increase in non-volatile carbon so that a high carb.–ash ratio is obtained for material of low fuel ratio.

The conclusion thus arrived at is that there is little difference between the ultimate products of deterioration of torbanite, irrespective of the quality of the original minerals, as all these themselves are deteriorated products from a common parent substance of high fuel ratio.

During the stages of deterioration it is probable that depolymerization processes play a big part in the chemistry of the kerogen molecules.

Such natural deterioration offers some striking parallelisms to certain cracking reactions. It appears that in both processes hydrogen is mainly removed, in the former probably by slow oxidation, in the latter by thermal dehydrogenation reactions. As a result the carbon contents of the volatiles show a steady increase up to a limit. Beyond this point in both cases carbon is separated, either as such or in the form of highly carbonaceous substances which rapidly decompose.

This theory of the deterioration of torbanite is substantiated by the following additional interesting facts observed by the author and recorded in his experimental work.

(*a*) Enormous difficulty is found in satisfactorily separating particular samples of torbanite into appreciable proportions of high- and lower-grade material by such physical means as specific gravity separation by float-and-sink methods, dry particle separation by screening, and various methods of hydraulic classification—*i.e.*, the ash associated with the mineral is mainly inherent.

(*b*) During carbonization, which can be considered to be a particular form of rapid artificial deterioration of torbanite at elevated temperatures, leaving spent torbanite as the final deteriorated product, an increase of fixed carbon is invariably observed, the important point being that material of high fuel ratio exhibits the increase of fixed carbon content to a higher degree than material of low fuel ratio, just as would be expected from the theory expounded.

(*c*) A similar increase in fixed carbon content is obtained in the spent torbanite after pressure extraction with a solvent. This process is also analogous to deterioration. Even solvent extraction under atmospheric conditions with pyridine gave a residue with higher fixed carbon than the original torbanite. The increase in this case was marked, although only a small extract was obtained.

(*d*) The carbonization of a weathered low-grade sample shows a lower yield of oil than freshly mined material as a result of preliminary deterioration. This tends to confirm the theory that during the deterioration of material of low fuel ratio, the hydrogen in the volatiles is attacked to a more appreciable extent than the carbon, leaving the latter behind in a non-volatile form.

(*e*) Irrespective of the quality of the original raw torbanite, the composition of the mineral matter associated with the organic material, as shown by the composition of the ash, is reasonably uniform.

(*f*) The ratio of yield of oil to yield of liquor of decomposition decreases considerably with decrease in fuel ratio, showing that during the stages of deterioration, water is definitely a degradation product, caused probably by the oxidation of hydrogen. On carbonization of the torbanite the " water of decomposition " distilled off can actually be regarded as the " water of deterioration " of the original high-grade mineral.

(*g*) The quantity of gas evolved from low-grade material is large in relation to the oil yield compared with that from the higher-grade mineral. This fact also suggests deterioration of rich torbanite to poorer grades.

(*h*) The general conclusion from a consideration of the distillation characteristics of the crude oil obtained from varying grades of torbanite is that they differ more in degree than in kind.

(*j*) The same general trend in the course of evolution of oil, course of evolution of liquor, and course of evolution of gas, irrespective of the class of torbanite retorted, leads to the firm conclusion that the different grades of torbanite are not as different as they appear, but are essentially the same parent substance manifesting itself in different stages of deterioration.

From the classification data given in Table V it appears that the material from the Giesecke Adit has deteriorated considerably as compared with that from the Carlis' or Troye Adits.

The formulæ derived in Table VI undoubtedly show that the mineral is extremely complex and variable according to its origin, and for this reason kerogen cannot be considered to be a definite chemical compound, but made up of molecules of various sizes and types, so that the oil-yielding portions of shales from different areas appear chemically unlike.

In a discussion on Scottish shale, Mill [24] regards the empirical formula for the total organic matter as the composition of the kerogen molecule. The tentative formula quoted is $n(C_6H_{10}O)$, n being undoubtedly large. It is debatable, however, whether Mills is entitled to regard all the organic constituents as taking part in the production of the volatiles. It is felt that the term "kerogen" should be applied only to that part of the organic matter in the material which gives rise to the oil, gas, and liquor.

Nitrogen apparently exists in combination with the oil-yielding matter, and as it is seldom considerably above 1 per cent. in these days of cheap synthetic ammonia processes, it is unlikely that ammonia recovery from shale carbonization as ammonium sulphate can be made a profitable proposition.

The sulphur found in torbanite may be partly adventitious, due to the presence of inorganic matter associated with the seam—*e.g.*, small quantities of pyrite (FeS_2), possibly pyrrhotite (Fe_6S_7 to $Fe_{11}S_{12}$), and gypsum ($CaSO_4,2H_2O$). In fact the characteristic structure of pyrite crystals can often be distinguished very clearly with the naked eye on torbanite samples taken especially near the division of the torbanite and coal layer associated with the seam, as adventitious mineral matter occurs most frequently at this point. The remainder of the sulphur is inherent in the mineral—*i.e.*, chemically combined in the organic matter.

The data given in Table IV for extraction of Transvaal torbanite by organic solvents at atmospheric pressure show that this method does not appear promising for successful operation, especially as the tests were carried out on the richest type of material. The extracts in no manner resembled the products of carbonization, and can therefore not be classed as oils in the accepted terms of the word. It is probably more correct to consider that the mineral contains a certain proportion of soluble bitumen, but little or no oil as such.

The effect of size of material, temperature, maximum pressure, and duration of pressure extraction of a particularly high-grade Transvaal torbanite has been discussed fully in the experimental section.

A glance at the results of pressure extraction of Transvaal torbanite, showing total extract on D.A.F. basis and as a percentage of the yield obtained by retorting, indicates that at high pressures, but moderately low temperatures, solvent extraction of torbanite gives a yield that offers satisfactory possibilities of application, especially if the extract were found to be particularly suitable, as such or slightly modified, for some specific purpose.

Kerogen is undoubtedly composed of large and complex molecules, as is also the bitumen formed in the first stage of decomposition. In general, the stability of organic molecules under the action of heat is in inverse proportion to their size and complexity. At high temperatures, therefore, the large molecules, firstly of kerogen and then of bitumen, decompose, break down, or crack into smaller, simpler, and more stable molecules. The retorting of oil shale is thus essentially pyrolysis of heavy and chemically complex bitumen, giving more simple molecules of gaseous, liquid, and solid hydrocarbons and their derivatives, which are more or less similar to those derived from oil-well petroleum.

In the author's own experimental work it was shown that the kerogen in Transvaal torbanite is practically insoluble in organic solvents at atmospheric pressures and can therefore be classed as an insoluble asphaltic pyrobitumen. On heating, the material becomes largely soluble. The exact nature of the change of pryobitumen to soluble bitumen is not known, but complete or partial depolymerization is suggested.

One concludes, therefore, that the extracts obtained by the author are the complex bitumens which are the first products formed in the destructive distillation of torbanite and are the parent substance for the further formation of oil, gas, and fixed carbon. During destructive distillation, however, both steps generally occur side by side, so that the bitumens cannot be retained without at least partial decomposition. On the other hand, in solvent extraction work at high pressures but moderate temperatures these primary bitumens can be isolated with little formation of secondary products beyond the small quantity of gas referred to previously.

The rate of conversion of pyrobitumen into bitumen appears to become vigorous at about 300° C. This applied to a high-grade torbanite.

From the author's experimental study of the progress of carbonization, it is interesting to see that this same temperature (300° C.) was found to be the decomposition point of rich torbanite. Poorer-grade torbanite, however, was found to decompose at a higher temperature (350° C.).

The author's observations on the effect of temperature of evolution on oil composition are confirmed with oil shales by Gavin,[2] who points out that the first distillates produced are not appreciably lighter in character than the oils produced at higher temperatures. The heavy bitumen first produced is decomposed into a mixture of light and heavy products. Gavin [2] also comes to the author's conclusions that although there is a possibility of separating the fractions by fractional condensation, it is preferable to use bulk condensation, with subsequent fractional distillation of the bulk condensate.

The liquor, of yellowish colour and vile odour, was not examined fully during the foregoing tests, except that the yield of ammonia, free and total, as ammonium sulphate was determined during several carbonization runs (Table VII). These figures show that the liquor is valueless and cannot be considered to be anything but a waste product of the industry. It might, however, find some use as a fertilizer, but on the whole it is more of a liability than an asset, as it requires suitable disposal.

Nash [34] discusses fully the possible uses to which the gaseous products of shale carbonization might be put. The chief use is undoubtedly for heating the retorts in which the shale is carbonized.

Reference to Table VII shows that spent torbanite comprises from about 40 to 80 per cent. of the products of carbonization, depending on the quality of the torbanite. In the treatment of torbanite an obvious great mechanical problem is the handling and disposal of this large amount of product, unless some suitable industrial use for the material can be found.

As the spent shale contains a high percentage of fixed carbon (35–45 per cent. in the samples quoted in Table VII), and is an unavoidable product of all retorting operations, one use as a fuel is immediately suggested owing to the large amount of heat still available from the material. Especially does this matter deserve attention when Manfield,[35] in a description of the Henderson retort, states that spent Dorsetshire shale containing only 12 per cent. carbon has been successfully applied as a fuel to supplement gas firing. Gavin [2] states that the use of spent shale containing only 9–14 per cent. free carbon in conjunction with fixed gases for firing the retorts saves 50 per cent. fuel cost and 33 per cent. labour.

Spent shale actually supplies all the heat necessary in the Davidson process of retorting Esthonian shale. In this connection it is interesting to note that Nash [34] states that the Esthonian Shale Oil Co. utilize the spent shale for the manufacture of producer gas either alone or mixed with raw shale. The calorific value of this gas is 180 B.ThU./cu. ft., the gas yield varying from 900 to 4300 cu. ft.

The fact that the ash content of Transvaal spent torbanite is so high (about 50–58 per cent.) should not afford difficulty of combustion, as the author has found that the material burns fairly easily in the atmosphere.

Thomas [36] states that satisfactory experiments have been carried out on spent shale as a cheap non-conducting material for electrical purposes. Other applications include the moulding of spent shale for railroad ties. It is claimed that shale ties are strong and lasting, and have a certain amount of elasticity lacking in cement or steel ties.

If spent torbanite is utilized, for example, as a fuel, its ash could be made available for other applications.

Torbanite ash, being low in iron, calcium, and magnesium, and having a high fusion point, appears to be promising as a refractory material. The author's observation of high fusion point (above 1500° C.) agrees with one determination by Neufeld.[37]

Transvaal torbanite ash might conveniently be employed in the manufacture of bricks for constructional purposes. Thomas [36] states that the Colorado deposits of shale ash are very satisfactory for this purpose, the bricks having greater strength for size and weight than bricks from ordinary clay or slate. He is of the opinion that this point should give the shale brick a clear preference in an even composition, apart from possible lower cost of material for making bricks if already mined or crushed waste shale is used.

It is of importance to note that from tests on Scottish shale ash, Gavin[2] comes to the conclusion that the colloidal properties of silica in that particular shale have been destroyed by the heat of the retort ; consequently the spent shale has no value for making brick, even when it is oxidized, as the binding properties of the clay are entirely destroyed.

Shale clay could undoubtedly also be used for such products as tiles, etc.

Lutz,[39] in a discussion of Esthonian oil-shale products, comments on the greater resistance to cracking offered by shale-oil constituents as compared with those of mineral oil.

It is of interest to compare the total yield of petrol obtained by the author by cracking Transvaal torbanite oil with those obtained by others for shale oils from other sources.

Hall[40] states that a 50 per cent. recovery of gasoline is obtained by cracking Scottish shale oil.

Egloff and Nelson[41] obtained a total yield of 49·4 per cent. gasoline by combined topping and cracking of Esthonian shale oil (22·0 per cent. by topping and 27·4 per cent. by cracking the topped crude), and a yield of 42·7 per cent. total gasoline when the whole crude oil was cracked without previously removing the straight-run spirit.

The same authors report a yield of 52–56 per cent. gasoline by cracking Manchurian shale oil to obtain a residuum directly suitable for bunker fuel. When a non-residuum method of operation was used, 67·4 per cent. gasoline was obtained, accompanied by a gas formation of 726 cu. ft. per barrel of crude oil and coke production of 54·8 lb. per barrel.

ACKNOWLEDGMENT.

The author desires to express his thanks to the South African Torbanite Mining and Refining Co., Ltd., for very kind permission to publish the results of experimental work presented in this thesis.

Bibliography.

[1] Hall, (*a*) "Investigation of Commercial Coal Samples," *J. chem., metall. min. Soc. S. Africa*, Feb. 1932, **32**. (*b*) "The Specific Gravity Investigation of Coal Samples," *ibid.*, Feb. 1934, **34**.

[2] Gavin, "Oil Shale : An Historical, Technical and Economic Study," *U.S. Bur. Mines, Bulletin* 210, 1924.

[3] Bone, Pearson, and Quarendon, "Researches on the Chemistry of Coal. Part III. The Extraction of Coals by Benzene under Pressure," *Proc. Roy. Soc.*, 1924, **105**, A, 608.

[4] Fischer and Gluud, "Die Ergiebigkeit der Kohlenextraktion mit Benzol," *Ber.*, 1916, **49**, 1469.

[5] Bakes, "The Action of Solvents on Coal." Dept. of Scientific and Industrial Research, Fuel Research Technical Paper No. 37, London, 1930.

[6] Kurth, "Report of Tasmanian Shale Oil Investigation Committee." Tasmania Dept. of Mines, Hobart, 1933.

[7] Bleloch, "The Ermeio Torbanite as a Source of Oils in the Union of South Africa," *J. Inst. Petrol. Tech.*, 1931, **17**, 521.

[8] Stewart and Trenchard, "The Destructive Distillation of Oil Shale," *Amer. Chem. Abs.*, 1924, **18**, 1048.

[9] Finley, Horne, Gould, and Bauer, "Assay Report Studies of Ten Typical Oil Shales," *ibid.*, 1924, **18**, 3711.

[10] Karrick and Jakowsky, "Problems in the Production of Oil Shale," *ibid.*, 1920, **15**, 943.

[11] Petrick, "A Contribution to the Study of South African Oil Shales," *J. chem., metall. min. Soc. S. Africa*, 1936, **37**, 236.

[12] George, "Retorting Problems of Colorado Shales," *Amer. Chem. Abs.*, 1921, **15**, 3641.

[13] Cunningham-Craig, "The Torbanites of South Africa," *J. Inst. Petrol. Tech.*, 1930, **16**, 620.

[14] Gray-King, "Carbonization Assay Methods of Analysis of Coal." Physical and Chemical Survey of the National Coal Resources No. 7. H.M. Stationery Office, 1927, p. 17.

[15] Fischer and Schrader, *Z. angew. Chem.*, 1920, **56**, 172.

[16] Crum-Brown. See Steuart, "Oil Shales of the Lothians." Memoir Geol. Survey Scotland, 1912, 2nd ed.

[17] Stopes and Wheeler, "The Constitution of Coal (I and II)," *Fuel*, 1924, **3**, 3, 63, 97, 129.

[18] Esling, "Estimation of Sulphur by the Lamp Method," *J. Inst. Petrol. Tech.*, 1921, **7**, 83.

[19] A.S.T.M. Standards on Petroleum Products and Lubricants. Annual publication by American Society for Testing Materials.

[20] "Standard Methods for Testing Petroleum and its Products." I.P.T., 1935, 3rd ed.

[21] Spiers, "Technical Data on Fuel," 1930.

[22] Mills-Davies, "Outlook for the African Oil Cor." (Map), *S. African Min. Eng. J.*, 1920, **29**, (II), 9–10, No. 1484.

[23] du Toit, "Oil Shale in South Africa," *S. African J. Industries*, 1921, **4**, 346.

[24] Mills, "Destructive Distillation." Gurney & Jackson, London, 1892, 4th ed.

[25] Anderson and Henderson, "The Coals of Bengal and Japan," *J. Soc. Chem. Ind.*, 1902, **21**, 237.

[26] Pearson, "Some New Derivatives of Bituminous Coal," *ibid.*, 1923, **42**, 68T.

[27] Illingworth, "Researches on the Constitution of Coal." Colliery Guardian Co., London, 1922.

[28] Meyer, *Braunkohlenarchiv*, 1927, **18**, 1.

[29] Szilard, *Chem. Ztg.*, 1929, II, 677.

[30] Franks, "Theory: Origin of Oil Shale," *Chem. & Met. Eng.*, 1921, **25**, 731, 778.

[31] McKee and Lyder, "Thermal Decomposition of Oil Shales: Kerogen—Intermediate Bitumen—Oil," *J.I.E.C.*, 1921, **13**, 613.

[32] Franks, "Studies in Colorado Shale Oils," *Chem. & Met. Eng.*, 1921, **24**, 561.

[33] McKee, "Oil Shales." A.C.S. Monograph Series, New York, 1925.

[34] Nash, "The Gaseous Products of Shale Retorting: their Composition and Possible Utilization," *J. Inst. Petrol. Tech.*, 1930, **16**, 626.

[35] Manfield, "Oil Shales, with especial reference to those of the Dorsetshire Kimmeridgian Series," *ibid.*, 1916, **2**, Pt. 7.

[36] Thomas, "Possible Uses of the Spent Shale from Oil Shale Operations," *Chem. & Met. Eng.*, 1921, **24**, 389.

[37] Neufeld, "Torbanite Carbonization and Products in South Africa," *J. chem., metall. min. Soc. S. Africa*, 1931, **32**, 24.

[38] Sexton, "Fuel and Refractory Materials." Blackie and Son, London.

[39] Lutz, "Der Estländische Brennschiefer—Kukersit: Seine Chemie, Technologie and Analyse." Tartu, 1934.

[40] Hall, "Cracking Scottish Shale: 50 per cent. Gasoline Yield Recovered from Shale Oil," *Petrol. World*, 1918, **15**, 337.

[41] Egloff and Nelson, (*a*) *Oil Gas J.*, 1933, **31**, (34), 14; (*b*) *J. Soc. chem. Ind.*, 1932, **51**, 326T.

ESTIMATION OF UNSATURATED HYDROCARBONS BY BROMINE ADDITION.*

By S. J. Green, B.Sc., Stud.Inst.Pet.

In the estimation of unsaturated hydrocarbons by halogen absorption many factors have been shown to affect the result, such as the time of reaction, temperature, ratio of reagent to hydrocarbon, the solvent used, the presence of catalysts and particularly the presence of oxygen. On the other hand, the type of olefine is equally important, since both the position of the double bond and the types of other groups present in the molecule can influence the rate of reaction. It is well known, for example, that the proximity to the double bond of electron attracting groups, such as the carboxyl, halide, and phenyl groups, is responsible for the low bromine values found for certain compounds. These effects are particularly great for bromine absorption as compared with iodine absorption, but since unsaturated compounds containing such groups are relatively rare in mineral oils, and because of the greater rate of reaction of bromine than iodine and iodine monochloride with olefines, bromine addition is, on the whole, preferable in the estimation of unsaturation in these oils.

The McIlhiney method [1] has the advantage of measuring the substitution as well as the addition value for oils, but Francis [2] has developed the potassium bromide–bromate method, in which any substitution is largely eliminated by the evolution of the bromine slowly in the reaction mixture. Cortese [3] has shown that this method is accurate for straight-chain compounds but only approximate for cyclics. Manning and Shepherd [4] have found the bromide–bromate estimation to give satisfactory agreement with their own acid-absorption method for the lower-boiling fractions of a refined oil, but obtained values too high for the heavier fractions, which was due, they stated, to the presence of toluene and xylene in these fractions. In 1935 Mulliken and Wakeman [5] studied the limits of the Francis method, and found it could be used accurately for most straight-chain olefines, but for the cyclic compounds and the alkadienes the accuracy was sufficient only to determine the number of double bonds. In their determinations these workers used volumes of brominating reagents in small excess of the calculated amounts required. Thomas, Block, and Hoekstra [6] have confirmed Mulliken and Wakeman's work and have modified the Francis method by repeated cooling of the reactants at several stages. Results for highly branched unsaturateds and polymers were found by Buc [7] to be too high, and he modified the Francis method by introducing excess potassium bromide, which modification he found to give results nearer the theoretical. In further modifying the method of Buc by titrating the unsaturateds direct with the bromide–bromate solution, Lewis and Bradstreet [7] eliminated the trial titration and limited the excess reagent to 1 c.c. They found it unnecessary to use a cooling mixture, but found that the bromine number of *iso*butylene increased with increasing excess of reagent, whilst erratic results were also obtained with tetra- and tri*iso*-butylene. These workers

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* Paper received 21st January, 1941.

found also that *iso*amyl mercaptan, benzyl mercaptan, amyl mercaptan, and *iso*amyl disulphide lowered the bromine number of di*iso*butylene, but that certain catalysts such as the salts of silver, mercury, zinc, and uranium prevented this.

In the present work, while estimating the heptene content of simple heptene–heptane and heptene–toluene mixtures by the normal Francis method[2] carried out at 0° C., by adding successive small volumes of bromide–bromate reagent and assuming a light yellow coloration of the reaction solution to indicate completion of the reaction, it was found that considerable error was sometimes involved. An inhibition effect apparently occurred when low concentrations of free bromine were present, because this light yellow colour of the reaction solution even after vigorous shaking did not necessarily indicate the complete bromination of the unsaturateds. Using known mixtures, a light yellow colour sometimes remained after several minutes' vigorous shaking with theoretically insufficient bromide–bromate solution.

An attempt was made to eliminate this error by modifying the Francis method and adding a volume of the bromide–bromate reagent certain to be in sufficient excess of the theoretical requirements. For this purpose, of course, a knowledge of the nature of the unsaturateds present is required. The effect on the bromine number of the volume of bromide–bromate reagent used was therefore investigated for several unsaturated hydrocarbons, and this modification of the Francis method was compared with the method of Lewis and Bradstreet.

EXPERIMENTAL.

Method Investigated.

25 c.c. of pure carbon tetrachloride were cooled in a glass-stoppered flask to 0° C. for 5 minutes and about 0·8 gm. of the sample introduced by volume or from a weighing pipette. The bromide–bromate solution sufficient to be in excess was then added, and a volume of 10 per cent. by volume sulphuric acid equal to half the volume of the former reagent. The flask was then shaken vigorously for 2 minutes, using a seconds-clock, and cooled before removing the stopper. A further 3 c.c. of sulphuric acid for every 10 c.c. of bromide–bromate reagent taken were added. After adding 5 c.c. of potassium iodide the liberated iodine was titrated with sodium thiosulphate in the usual way. The bromine number was calculated as shown :

$$\text{Bromine Number} = \frac{8(B - X)N}{W},$$

where B = c.c. thiosulphate solution for appropriate blank determination, X = c.c. of thiosulphate for determination, N = normality of thiosulphate solution, and W = gm. sample taken.

Modified Lewis and Bradstreet Method.

20 c.c. of 10 per cent. sulphuric acid were introduced into the flask with the cooled solvent and hydrocarbon. The bromide–bromate was run in from a burette in small quantities and with vigorous shaking after each

addition. When a yellow colour remained after 3 minutes' shaking a further 0·5 c.c. was run in and the solution titrated with sodium thiosulphate as usual.

The sulphuric acid was not saturated with potassium bromide as used by Lewis and Bradstreet, but all the reagents were as described by Francis.[2] Mixtures of technical heptene (bromine number 165) with toluene and with heptane were examined. The results obtained by using 30 c.c. of the bromide–bromate reagent and by the Lewis and Bradstreet method are shown in Table I.

TABLE I.

Heptene Mixtures.
(*Taking* 0·6–0·7 *gm. of hydrocarbon mixture in each case.*)

Theoretical bromide–bromate solution for 0·7 gm. *n*-heptene = 29·1 c.c.

Mixture.	% Heptene in mixture.	Approx. excess of bromide–bromate (estimated).	% Heptene found using 30 c.c. re-agent in each case.	% Heptene found by L.B. method.
Heptene–heptane mixtures	100·0	0·9 c.c.	102·0	102·1
	80·6	7·7 ,,	78·8	80·0
	60·0	12·5 ,,	58·3	60·1
	36·8	19·2 ,,	34·7	36·0
	19·5	24·2 ,,	18·2	20·0
	10·0	26·0 ,,	10·0	9·8
Heptene–toluene mixtures	100·0	0·9 c.c.	102·0	102·1
	81·6	7·7 ,,	77·8	80·5
	62·47	12·0 ,,	59·4	62·0
	41·69	15·0 ,,	40·0	41·0
	21·42	22·0 ,,	23·4	21·5
	10·59	25·0 ,,	11·9	11·9

The magnitude of the excess of bromide–bromate above the theoretical in the estimation of heptene has little influence on the result, although the titration method of Lewis and Bradstreet, where the excess reagent is limited to less than 1 c.c., is more accurate.

The effects of varying the excess of the bromide–bromate reagent for estimation of oleic acid in *n*-heptane, of *cyclo*hexene, and of technical nonene were observed. The oleic acid used was purified by two vacuum distillations and equilibrium freezing at 14° C. Pure *cyclo*hexene (b.pt. 83° C.), was used, obtained by repeated distillation from fresh sodium after standing with sodium for seven days.

Examples of the results obtained are shown in Table II.

For oleic acid and nonene the effects of excess bromide–bromate are quite small, and negligible for most purposes, but although the results for *cyclo*hexene are inconsistent, the general trend is the suppression of bromine absorption with increased reagent excess. In the latter case the use of larger volumes of carbon tetrachloride to eliminate any effect due to the distribution coefficient of bromine in the two phases, reagent phase and solvent phase, and the use of chloroform and heptane as solvents does not appreciably affect the results. Saturated hydrocarbons tested do not absorb bromine under the conditions of the reaction, although in one case for a mixture of 1 c.c. *m*-xylene, 1 c.c. toluene, and 1 c.c. benzene, a small absorption was observed.

TABLE II.

Sample.	Volume of bromide–bromate reagent used.	Bromine number.	
		Found.	Theoretical.
1 c.c. technical nonene	30 c.c.	109·9, 109·0	126·9 for pure nonene
,, ,, ,, ,,	25 ,,	110·2	
1 c.c. benzene	10 ,,	0	0
1 c.c. toluene	10 ,,	0	0
1 c.c. *m*-xylene	10 ,,	0	0
1 c.c. toluene 1 c.c. xylene 1 c.c. benzene } mixture	10 ,,	0·4	0
Heptane	10 ,,	0	0
Hexane	10 ,,	0	0
*iso*Octane	10 ,,	0	0
1 c.c. *cyclo*hexene	Titration method	193, 193·4	195·1
,, ,,	2 c.c. excess	195·8	195·1
,, ,,	,, ,,	195·7 *	195·1
,, ,,	7·6 c.c.	182·3	195·1
,, ,,	17·6 ,,	186 †	195·1
,, ,,	17·4 ,,	180·8	195·1
,, ,,	32·7 ,,	190·0	195·1
Synthetic mixture 8·29% oleic acid in heptane	2 c.c. excess	4·7	4·73
	8 ,,	4·72	

* Chloroform as solvent.
† Equal volumes of aqueous reagents and carbon tetrachloride.

Estimation of Unsaturateds in Complex Mixtures.

In the estimation of unsaturateds in benzole the Francis method was found to give sometimes low results, apparently due to the inhibition effect described above, and when this method was modified by introducing somewhat larger excesses of bromide–bromate reagent the bromine value was increased accordingly. Although the actual percentage of the unsaturateds present was unknown, the Lewis and Bradstreet method appeared to be more accurate, because it gave perfectly consistent results reasonably higher than the lowest value obtained using the ordinary Francis method.

A benzole fraction 90–130° C. was examined. The sulphur content was 0·5 per cent., but most of this could be assumed present as carbon bisulphide and thiophen. Since the small inhibition effect observed using the Francis method was not overcome by the use of 1 c.c. of a 10 per cent. uranium acetate or zinc sulphate solution, which substances were shown by Lewis and Bradstreet to suppress the inhibition effect due to sulphur compounds, the observed effect was probably not due to such compounds. The effect was not found at all, however, with the titration method developed by these workers. With increasing excess of the reagent the bromine number was found to increase to a constant but inordinately high value.

Using the ordinary Francis method the low value of 21 was obtained when there was still an apparent excess of reagent even after shaking for 4 minutes. In Table III is shown the results using different excesses of bromide–bromate.

TABLE III.

Bromine Numbers on Benzole Fraction.

1 c.c. (0·808 gm.) benzole in each case.

Excess bromide–bromate reagent.	Bromine number.
Titration method	27·0, 27·0
1 c.c.	26·4, 28·0
4 ,,	30·4
14 ,,	35·6
19 ,,	36·0
24 ,,	36·1
30 ,,	36·0

Although theoretically the modified Lewis and Bradstreet method used does not remove the possibility of under-estimating the bromine absorption as described above for the Francis method, in practice this error does not arise, due perhaps to the greater time of reaction and the addition of the reagent in small quantities.

For the estimation of the unsaturateds in hydrocarbon mixtures, apart from the sources of error indicated above there is also the possibility of error in the estimation of the molecular weight and the possible presence of diolefines. Within certain limits the molecular weight can be estimated from the boiling range by comparison with the curve of average boiling points against molecular weights for the mono-olefines. Above 140° C., however, the limits are very wide and such estimation must be considered quite unsatisfactory.

The unsaturated hydrocarbons were isolated from the 90–130° C. benzole fraction by bromination, distillation from bromo-compounds to 127° C. followed by debromination with zinc at 30° C. and redistillation. The product was distilled again and fractionated. The molecular weights of the fractions were determined by the cryoscopic method and also estimated from the molecular weight–average boiling point curve for the mono-olefines. All the evidence indicated that the unsaturateds present in the

TABLE IV.

Boiling range unsats., ° C.	Refractive index, n_D^{20}.	Bromine number of unsats.	Molecular weight.		No. of double bonds to nearest unit.
			Found.	Estimated.	
80–132	1·4400	140·0	96	105	1
132–139	1·4456	59·0	119·7	127	1
139–145	1·4502	55·0	119·1	137	1
above 145	1·4483	33·0	123·7	above 140	2

benzole were highly branched chain mono-olefines, although the possibility of a change in the constitution during the isolation is by no means small. A trace of aromatics was found in each fraction of the isolated unsaturateds by nitration, reduction of nitro-compounds, diazotization, and coupling with β-naphthol; however, there was nothing to show whether the aromatics were present as impurities or combined in the unsaturated compounds. The bromine numbers of the unsaturated fractions were found by the titration method, but did not show favourable agreement with the molecular weights for the higher fractions.

The results are recorded in Table IV, together with the molecular weight estimated from the mono-olefine curve of average boiling point against molecular weight.

CONCLUSION.

An inhibition effect which might be a potential source of error was observed in brominating heptene, nonene, and a benzole fraction by the Francis method, but this did not occur when using the Lewis and Bradstreet titration technique. The bromine number can be used for comparative purposes as a measure of unsaturation in complex hydrocarbon mixtures, but the conditions of the reaction should be specified precisely. The Lewis and Bradstreet method is recommended as most satisfactory, however. For the estimation of the actual percentage of unsaturateds the bromine absorption method is unreliable unless the molecular weight and type of the olefines present can be determined or estimated with accuracy.

References.

[1] McIlhiney, *J. Amer. Chem. Soc.*, 1899, **21**, 1084.
[2] Francis, *Industr. Engng Chem.*, 1926, **18**, 821.
[3] Cortese, *Rec. trav. Chim.*, 1929, **48**, 564.
[4] Manning and Shepherd, *D.S.I.R.*, **28**, 1930.
[5] Mulliken and Wakeman, *Industr. Engng Chem. Anal.*, 1935, **7**, 59.
[6] Thomas, Block and Hoekstra, *ibid.*, 1938, **10**, 153.
[7] Described by Lewis and Bradstreet, *ibid.*, **12**, 387.

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ABSTRACTS.

AUTHOR INDEX.

The numbers refer to the Abstract Number.

The original papers referred to in the abstracts marked with an asterisk may be borrowed by Members from the Institute Library.

Geology and Development.

101.* Sink-holes Important Feature in Arbuckle Dolomite. Anon. *Oil Gas J.*, 12.12.40, **39** (31), 21.—Sink-holes are common in the Arbuckle lime. In the Silica field of Kansas 36 out of 70 dry holes were due to sink-holes of which there is apparently no surface indication. Their barrenness is due to compacting overlying detritus preventing leaching and subsequent porosity. The fill of these sinks ranged from 14 to 163 ft. In some cases barren sinks were completely surrounded by productive wells, and one sink was only 106 ft. from the nearest producer. The sink-holes occupy only a small area, and rarely exceed the 10 acres allotted to a well. G. D. H.

102.* Wade City–Orange Grove May See Multiple Sand Drilling. N. Williams. *Oil Gas J.*, 12.12.40, **39** (31), 26.—This is already one of the most important producing reserves of the Lower Texas Gulf Coast. There is much productive acreage to be exploited, and some of the sands seem capable of multiple development. A well 1½ ml. south of Wade City and 1½ ml. south-west of Orange Grove may be a new pool or an extension of the Wade City–Orange Grove area. 3 ml. south-east of Orange Grove a new area has been opened at 5139–5143 ft., and a test 3 ml. east of Orange Grove has shown oil and gas at 4863–4875 ft.

Wade City and Orange Grove are separated only by a subsurface fault, the former being on the upthrown north-west side. The throw is 250–300 ft. Wade City production covers 1400 acres, and Orange Grove 600 acres. Wade City began producing with gas-distillate in July 1939 at 4800 ft. in the Pundt sand. Crude oil was found in February 1940 in the McNeil sand at 4900 ft. Orange Grove production began in May 1940 in the Blaschke sand at 5100 ft. This has given most oil, although the Pundt 50 ft. below has given some. Wade City has 70 producing wells, Orange Grove 19.

Wade City gives oil from the Pundt (gas and distillate), Bierstadt, and McNeil sands, whilst the Alice at 5000 ft. has favourable showings. In addition, there are lesser sands and lenses, mainly with gas. The McNeil is the most consistent sand, the others being very variable. Orange Grove has Blaschke, Pundt, and Stillwell (distillate) sands. All the known sands of these fields are Basal Frio and Upper Vicksburg. G. D. H.

103.* Illinois Activity Concentrated on Eastern Side of State. H. F. Simons. *Oil Gas J.*, 19.12.40, **39** (32), 15.—Although Illinois activity has declined generally in recent months, there has been an increase in activity in White, Wabash, Edwards, Gallatin, Franklin, Hamilton, and southern Wayne counties. The fact that White County lies in the deepest part of the Illinois basin discouraged development in the early days. Most of the fields developed in this region are small, for, although there are large anticlines, oil is confined to rather small structures on these larger features. There are several producing sands in this area—East Calvin has six horizons. Except for the McClosky, all the producing horizons at East Calvin are in the Chester series, and include the Tar Springs, Cypress, Benoist, Paint Creek stray, and Aux Vases sands.

The first production in this eastern area was at Aden in 1938. The discovery wells in South-eastern Illinois, their date of completion, depth, producing horizon, and initial production are tabulated. The production, number of wells, etc., for the fields on 1st November, 1940, are compared with the data for 1st January, 1940, and the total output is seen to be nearly quadrupled.

The McClosky wells generally have a higher initial output, but the sand wells are longer-lived. G. D. H.

104.* Argentina's Crude Production up 10% in 1940. R. Silveyra. *Oil Gas J.*, 26.12.40, **39** (33), 78.—Mendoza province has given 90% of the 1,479,745 brl. increase in Argentina's oil production in the first nine months of 1940, and the country's total for 1940 will probably be 20,476,130 brl.—10% above 1939. Comodoro Rivadavia has given 73% of the total for the first nine months of 1940.

At Barrancas a 285-brl. well was brought in at 7382 ft., and this discovery was confirmed by a well 4·35 ml. to the north-west, which came in at 1260 brl./day. Tupungato is now under regular exploitation, and the discovery well has averaged 2190 brl./day during its first eleven months.

Tables show the production during the first nine months of the years 1931–1940, the production of the various regions during the first nine months of 1939 and 1940, the capacities of the refineries in 1940, the amounts of the refinery products and home consumption. G. D. H.

105.* War Exerting Strong Influence on Venezuelan Oil Industry. L. Herrera-Figueredo. *Oil Gas J.*, 26.12.40, **39** (33), 80.—Venezuela's 1940 production is estimated at 186,000,000 U.S. brl., 10·6% less than the 1939 figure, and the exports are probably 161,000,000 brl., or 16·2% less than in 1939. The refinery processing of 17,600,000 brl. of crude in the first nine months of 1940 was 97% above the figure for the same period in 1939.

New fields—San Joaquin, El Roble and Santa Ana—have been brought under development in 1940. San Joaquin had six producing wells up to 1st October, 1940, which averaged 710 brl. of oil/day each. El Roble has two wells which average 2000 brl./day each. Santa Ana has two wells, and lies 25 ml. north-west of Oficina. No. 1 Leona in Anzoategui came in at 1000 brl./day in October. So far tests in Guarico have been unsuccessful. A well has been completed successfully at Los Caritos, Monagas.

The production by fields and companies is tabulated for the first nine months of 1939 and 1940. G. D. H.

106.* Peru's Agua Calientes Field is Developing Normally. L. P. Stockman. *Oil Gas J.*, 26.12.40, **39** (33), 104.—Four wells have now been drilled on the Agua Calientes structure. The first was stopped at 3100 ft. at the beginning of 1939, having failed to give production, although oil and gas showings had been noted. The second gave 750 brl./day of 45° oil at 1175 ft. in February 1939, and was shut in later. The third well farther down the structure failed to produce at 3000 ft., whilst the fourth came in at 200 brl./day at 1180 ft.

The structure has a closure of 3600 ft. Production is from a Cretaceous sand. There is an associated structure to the south-east and extensive seeps occur 20 ml. to the south-west. The crude is of a paraffin base.

Some indications are given of the practical problems involved in the development of this difficultly accessible region. G. D. H.

107.* Four Areas in Brazil Warrant Petroleum Investigation. F. N. Bosco. *Oil Gas J.*, 26.12.40, **39** (33), 120.—(1) The coastal belt about 25 ml. wide, and running for 140 ml. north of Bahia, consists mainly of downfaulted, chiefly lacustrine Cretaceous beds dipping seaward. In the north they are overlain unconformably by Tertiaries, calling for geophysical work. A well drilled slightly north-west of Bahia, near the Lobato seep, had an estimated production of 20–50 brl. of oil/day at 1150 ft. A second well 31 ft. deeper may give 100 brl./day.

(2) The Parana basin is a structural syncline 800 ml. from north to south and 600 ml. wide. It has probably 5000 ft. of Cretaceous, Permo-Triassic, and Devonian beds, including volcanics and intrusives, resting on pre-Cambrian crystalline rocks. Some 38 exploratory wells have failed to find commercial oil accumulations. The limited number of seeps and the igneous activity are thought to be unfavourable features for oil in this region, but deep tests are required for conclusive information.

(3) The Central Amazon valley is also a structural syncline. It is 800 ml. wide, and contains Tertiary, Mesozoic, and Palæozoic beds with several unconformities.

(4) The territory of Acre adjoining Peru is potentially productive. There are

favourable structures which have, however, only been partly tested by single wells. Tertiary, Cretaceous(?), and Palæozoic beds are present.

Stratigraphical tables are given for the first three of these potential oil regions, which are placed in order of their probable importance. G. D. H.

108.* Alaska Devoid of Active Drilling Operations. L. P. Stockman. *Oil Gas J.*, 26.12.40, **39** (33), 140.—The Salmon Creek structure on the eastern side of the Alaskan Peninsula was tested to a depth of 7596 ft., and the well was then abandoned. This failure has caused the abandonment of plans for drilling on four similar structures. It is uncertain whether this latest well stopped in the Lower Jurassic or in the Upper Triassic. The beds proved thicker than was expected. The well-defined Salmon Creek structure covers 20,000 acres.

In 1922 two wells were drilled on the Pearl Creek structure, 25 ml. north-west of Salmon Creek.

There are prominent oil seeps in the Cold Bay region, around Aniakchak Bay and Chignik. In the Cold Bay region they are along anticlinal lines. Both the Salmon Creek and Pearl Creek anticlines have strong seepages.

Apparently the Pearl Creek wildcat stopped 1000 ft. above the Jurassic–Triassic contact. The Jurassic and Triassic rocks appear conformable, but in the absence of fossil evidence, determination of the exact contact is difficult. G. D. H.

109.* Mexico's Production Shows Modest Gain. Anon. *Oil Gas J.*, 26.12.40, **39** (33), 152.—Mexico's production for 1940 is estimated at 43,470,000 brl., an increase of about 1% over 1939. However, the yield for the first nine months was 6% above that for the corresponding period of 1939. Poza Rica has provided about 64% of the 1940 total. Tables show the monthly production and exports up to September 1940.

In September there were 881 producing wells, 95 more than in September 1939. The four main pipe-line systems total 724 ml. in length and handle an average of 112,865 brl. of oil/day, whilst the seven refineries have a daily capacity of 99,800 brl., with cracking facilities for 18,830 brl./day.

The effect of the war on Mexico's oil exports is discussed. G. D. H.

110.* Michigan Getting First Real Deep Exploration. Anon. *Oil Wkly*, 9.12.40, **100** (1), 24.—For fifteen years Michigan drilling has been mainly to the Traverse and Dundee limestones. Little oil has been obtained from other formations, and the few scattered deeper tests have been dry. The Traverse is the main producer in the west, and the Dundee in the centre of the State. Production has fallen off rapidly during 1940.

A deep well is under way to test formations below the Sylvania in the centre of the State. Oil and gas shows are known in the Niagaran, and the Trenton produces in Monroe county. Altogether 15 tests have gone to the Silurian and Ordovician outside the central basin province. The sites and depths of a number of deep tests are listed and some details given of the nearest one to the new well. This new well showed oil in the Sylvania and Monroe, and details are given of the drilling. The Salina of the Silurian was met at 5480 ft. Gas trouble developed at 7368 ft. The Niagaran is expected at 8000–8300 ft., the Trenton possibly at 9200–9500 ft. and the St. Peter at about 10,000 ft.

At Deerfield are 15 Trenton wells. The St. Peter has shown oil on the edge of the central basin.

A generalized geological column of Michigan is included, and there are tables of production, and distribution of the geological formations of the deep discoveries and new producing levels uncovered in the portion of North Texas since 1936, and Michigan wildcat results since 1931. G. D. H.

Drilling.

111.* Temperature Surveys in Oil Wells. C. V. Millikan. *Petrol. Tech.*, Nov. 1940, A.I.M.M.E. Tech. Pub. No. 1258, 1–8.—The thermometers should read to 0·2° F. or less and have a low thermal lag. Vapour-pressure and expansion types are easily manipulated in the well, being self-contained, but the temperature record cannot be seen until the run is completed. The more sensitive electrical resistance type records at the surface, but requires more elaborate equipment.

If possible the part of the hole to be examined should be filled with liquid. The value of the temperature survey depends on the successful interpretation of the anomalies. The well should be in such a condition as to give the greatest temperature anomalies, and the run should be made not too soon after shutting in, but before temperature equilibrium has been attained. Stratigraphical determinations are possible from temperature surveys in certain well-developed fields. Gas sands can be picked up from temperature anomalies (lows), as can the gas–oil contact in a producing well. Thief formations, water sources, and casing leaks can also be determined. Cement setting behind casing can be located by the heat generated.

Examples are given of these various uses of temperature surveys, and the conditions for best results are described in each case. G. D. H.

112.* Fewer Casing Sizes in New A.P.I. Specifications. Anon. *Oil Wkly*, 9.12.40, **100** (1), 34.—Adoption of a new casing list, designed to reduce to 86 from more than 200 the number of sizes, weights, and grades of casing, was recommended to the American Petroleum Institute by its Division of Production Committee on Standardization of Oil Country Tubular Goods. The simplified list is being studied for final ratification.

The list was formulated and is being revised to contain all casing which may be required for drilling oil wells to any depth. Details of the sizes and grades recommended are presented.

In arriving at the 86 items in the list (4½-in. to 13⅜-in. range), an attempt was made to establish increments of about 1000 lb. in minimum collapsing pressures for the smaller sizes. The outer diameter increment for these sizes approximates ½ in. Between 4½-in. and 7⅝-in. sizes, the ½-in. is approximately maintained, whilst from 7⅝-in. to 11¾-in. sizes the 1-in. increment is the unit. In the larger popular sizes (8⅝-in. to 13⅜-in.), minimum collapsing pressure increments are smaller, ranging from 750 to 500 lb./sq. in. A. H. N.

113.* Slightly Less Drilling in 1941 Tentatively Indicated. Anon. *Oil Wkly*, 9.12.40, **100** (1), 38.—The tentative conclusion that a smaller number of wells will be drilled in 1941 than was accomplished in 1940 is based on information supplied by a group of oil companies, limited in number, yet constituting a fairly good cross-section of the industry.

The causes are briefly analysed. A. H. N.

114.* Recent Advances in Illinois Drilling Practices. B. De Laat. *Oil Gas J.*, 5.12.40, **39** (30), 45.—Drilling conditions in Illinois are distinctly different from those in most other fields in the U.S.A. The interval drilled by each bit rarely exceeds 300 ft. Thus, apart from the use of a great number of bits, drilling time is adversely affected. A simple apparatus which has been found useful in giving an index to the efficiency of drilling consists of industrial speed-indicating devices and revolutions counters. This equipment yields information concerning the number of revolutions of the bit needed to penetrate the various formations. From these data, the penetration index is obtained, which is the number of ft. cut/1000 revolutions of the rotary table, and appears to be an absolute term. Influence of various factors on drilling time is being studied individually.

If the weight on the bit can be kept in the neighbourhood of 3000 lb./sq. in. of bit, it has been found that drilling rates are greatly increased. In certain formations, doubling the weight increases the rate by 400%. Multiple or tandem drill-collars are used in experiments to increase drilling rates.

Mud problems peculiar to Illinois fields arise from the need to maintain delicate hydrostatic balance if loss of circulation is to be avoided.

Slim-hole drilling is reviewed. A. H. N.

115.* Applying Tool-Joints to Pipe at Remote Locations. N. Williams. *Oil Gas J.*, 12.12.40, **39** (31), 34.—Because of the advantages of being able to replace worn tool joints in the field, oil operators and manufacturers are developing tool joints which are not only simple in their application in the field, but also eliminate the chances of improper fitting which always exist when no mill or machine-shop facilities can be used

"Shrink thread" or "Shrink grip" joints are described which can be installed without the use of bucking-on machines. The joint is designed with an extra length of gripping surface to provide a greater bond with the pipe and further relieve the strain on the threads; it also contains an inner seal that fits against the end of the pipe. The latter not only serves as a fluid seal, but also as a definite reference mark in sizing the tool-joints. The tool-joint is screwed on until the shoulder hits the end of the pipe, this being the only position in which it fits.

As a result of these developments in design of tool-joints, their replacement has resolved itself mainly into a mechanical problem of cutting off the old tool-joints and screwing on the new.

Details of the operations involved in cutting the old tool-joint, straightening pipe, lubricating the threads, and screwing on the new joint are given in full.

A. H. N.

116.* Determining Properties of Rotary Drilling Fluids. H. F. Simons. *Oil Gas J.*, 12.12.40, **39** (31), 36.—Note is taken of the lack of instrumentation on most drilling rigs and consequent lack of control and efficient handling of mud. Where instruments of simple types are found, these are often used incorrectly and the results become unimportant and devoid of potential correlation and value.

The characteristics of the muds to be determined are density, viscosity, wall-building properties, thixotropy, p_H values, sand content, salinity, and particle size. Instruments and procedures for the determination of these properties are described, the determination of density, viscosity, and wall-building properties being studied in greater detail than the rest.

The correct use of the hydrometer, Marsh funnel, and Stormer viscometer is emphasized by quoting several instances and methods of abusing these instruments or following wrong procedures. No theoretical or analytical treatment is presented, as the paper deals primarily with the practical aspect of manipulating these instruments.

A. H. N.

117.* Evaluation of Rotary Drilling Lines. H. C. H. Darley. *J. Inst. Petrol.*, August 1940, **26** (202), 407–412.—A formula is derived for the work done, in ton-miles, by a drilling line per round trip in the rotary drilling system, and the formula is compared with the Hallam Anderson's formula as given by Saatjian and Mason and approved by the American Petroleum Institute. Readings made from the alignment chart of Saatjian and Mason are, on a broad average, 50% higher than those obtained from the author's curve. A. H. N.

118.* Control of Drilling Mud in the Field. H. C. H. Darley. *J. Inst. Petrol.*, October 1940, **26** (204), 457–474.—A study of the more fundamental properties of mud fluids is made and experiments are described to be carried out in the laboratory or in the field in order to control these properties and maintain them within the range in which they are of greatest use and importance. A. H. N.

119.* Further Investigations of the Properties of Clay Suspensions. G. D. Hobson. *J. Inst. Petrol.*, December 1940, **26** (206), 533–564.—The investigations deal with experiments on clay in connection with flow properties as exhibited by capillary and modified rotary viscometers. An interesting saw-tooth curve is obtained with the latter instrument when torque is plotted against angle of rotation. Correlations with results of other experimenters are made throughout the paper. A. H. N.

120. Patents on Drilling. D. Scaramucci. U.S.P. 2,223,388, 3.12.40. Appl. 28.10.39. Cementing head with a chamber adapted to receive the lower of two cementing plugs.

R. Schaedler. U.S.P. 2,223,389, 3.12.40. Appl. 20.9.39. Guy wire fastener.

W. Bendeler and P. Howard. U.S.P. 2,223,439, 3.12.40. Appl. 11.4.40. Well perforator using a cutter actuated by a cam.

E. V. Crowell. U.S.P. 2,223,442, 3.12.40. Appl. 14.8.39. Apparatus and method of cementing wells using cement discharge ports which may be opened to allow passage of successive volumes of cement.

L. F. Brauer. U.S.P. 2,223,509, 3.12.40. Appl. 24.5.39. Float-valve for use in a drill pipe assembly.

C. White and O. B. Graham. U.S.P. 2,223,651, 3.12.40. Appl. 1.8.40. Slush-pump valve.

R. R. Bloss. U.S.P. 2,223,779, 3.12.40. Appl. 24.5.40. Rotary drilling machine.

Hijman Lumberg. U.S.P. 2,223,789, 3.12.40. Appl. 3.1.39. Process for sealing water-bearing strata, using a solution of asphalt bitumen in an organic water-miscible solvent, and precipitating the asphalt by dilution with water.

H. T. Kennedy. U.S.P. 2,223,804, 3.12.40. Appl. 4.11.38. Method of sealing pervious strata in oil or gas wells.

R. A. Wilson. U.S.P. 2,223,839, 3.12.40. Appl. 30.9.38. Valve-in-head packer

W. L. Church. U.S.P. 2,223,984, 3.12.40. Appl. 16.11.39. Outside pipe cutter.

S. P. Hart. U.S.P. 2,224,120, 3.12.40. Appl. 21.7.38. Treatment of oil wells, for recovering lost circulation during drilling, with a casein plastic containing more than 5% by weight of casein. A. H. N.

See also Abstract No. 213.

Production.

121.* Phase Behaviour in the Methane–Propane–*n*-Pentane System. R. T. Carter, B. H. Sage, and W. N. Lacey. *Petrol. Tech.*, November 1940, A.I.M.M.E. Tech. Pub. No. 1250, 1–8.—The method of examining the phase behaviour of the methane–propane-*n*-pentane system is briefly described, and its limitations are indicated. The results obtained show that at 100° F. the critical pressures of the ternary mixtures are less than the critical pressure of the binary mixture the components of which exhibit the maximum divergence in critical temperature. However, the experimental work was not sufficiently complete to establish the critical behaviour with great accuracy, and it is possible that a small maximum might exist in the ternary region in the immediate vicinity of the methane-*n*-pentane axis. The measurements indicate a marked influence of the nature and amount of other components on the phase behaviour of these lighter paraffin hydrocarbons. These effects are appreciable in propane and *n*-pentane at a pressure of 500 lb./in.^{2}, and pervade the entire two-phase region for methane. This feature limits the usefulness of the equilibrium constants, for they are a function of the heterogeneous system rather than a quantity that is a function only of pressure and temperature. At pressures in excess of 1000 lb./in.^{2} the variations are such as to render the prediction of the phase behaviour of systems of this nature, from generalizations based alone on variations in pressure and temperature, of little avail. G. D. H.

122.* Volumetric Behaviour of Oil and Gas from the Rio Bravo Field. B. H. Sage and H. H. Reamer. *Petrol. Tech.*, November 1940, A.I.M.M.E. Tech. Pub. No. 1251, 1–12.—Samples of the gaseous and liquid phases of a field with a reservoir pressure of 4500 lb./in.^{2} and temperature of 250° F. were obtained from a production trap, and their volumetric behaviour and that of eight mixtures of the two was determined for pressures up to 5000 lb./in.^{2}, and temperatures from 100° to 250° F. Data about the chemical constitution and physical properties of the trap samples are given.

Tables give the compressibility factors for the gas sample, the specific volumes of the trap liquid sample, and the composition, specific volumes, and formation volumes of mixtures of the trap samples. Curves show the compressibility factor for the trap-gas sample, the specific volume of the trap-liquid sample near the bubble point, the effect of gas/oil ratio on formation volume and on bubble-point pressures.

An equation is given which approximately relates the formation volume of a heterogeneous mixture and the behaviour of the bubble-point liquid and the trap-gas sample. G. D. H.

123.* Equilibrium Constants for Hydrocarbons in Absorption Oil. C. E. Webber. *Petrol. Tech.*, November 1940, A.I.M.M.E. Tech. Pub. No. 1252, 1–11.—The absorption oil was a straw-coloured, steam-distilled oil of an initial boiling point of 300° F. *n*-Hexane, propane, *iso*butane, *n*-butane, *iso*pentane, *n*-pentane, ethane, and methane were the light hydrocarbons used. The apparatus in which equilibrium between the hydrocarbons methane to hexane and natural gas and a typical absorber oil for temperatures of 33–180° F. and pressures of 100–5000 lb./in.^{2} was determined, is described in detail, together with the experimental technique. The analytical results are tabulated, and the equilibrium constants are plotted against the pressure for temperatures of 100° and 180° F. Each of the K-value curves for all the hydrocarbons heavier than methane was found to pass through a minimum, and thereafter increase rapidly with increasing pressure. The pressures at the minimum K-values varied with the temperatures and for the different components, being lower for the heavier components. At the higher pressures the K-values for all the components approached unity, but there was a significant reduction in the rate of approach at 5000 lb./in.^{2}.

The K-values seem suitable for any multi-component hydrocarbon system up to 1500 lb./in.^{2}. Above 3000 lb./in.^{2}, the composition of the mixtures has a pronounced effect, and care must be exercised in applying these values to systems that differ considerably from that investigated. Absorber operations seem feasible, even above 2000 lb./in.^{2}, the optimum point with regard to oil circulation being at the pressure where the K-values are at a minimum for the given temperature. G. D. H.

124.* Study of Well Spacing, Sinclair–Moren Pool, Young County, Texas. M. G. Cheney. *Petrol. Tech.*, November 1940, A.I.M.M.E. Tech. Pub. No. 1253, 1–4.—The producing record of the Sinclair–Moren pool shows that under efficient operating conditions a single well may obtain normal oil recovery from an area of 100 acres or more, and this was where water drive was lacking, initial reservoir pressure only moderate, and the consolidated sandstone reservoir had an original permeability greatly reduced by lithefaction. The well penetrated the sand from 2512 to 2559 ft., and initially gave 12,000,000 cu. ft. of gas and 1100 brl. of oil/day. Two other wells did not find the sand, and a third was a poor producer. Ten years later the well had given 1,160,000 brl. of oil and was still giving 90 brl./day at 120 lb./in.^{2}. The drilling-in evidence is against its being a crevice well, and its good production was apparently due to the absence of nearby competitive wells for over ten years, to gas conservation by efficient production methods, and perhaps to the presence of 50 ft. of oil sand. At this time further wells were drilled which had good outputs. From a consideration of sand thickness and the well's output, this first well seems to have been draining about 100 acres effectively. Its gas/oil ratio was cut back strongly soon after its completion.

The record of this lease appears to demonstrate that the withdrawal of oil through closely spaced reservoir outlets has been at the expense of producing efficiency. G. D. H.

125.* Some Factors Influencing Recovery of Condensate in Recycling Operations. L. S. Reid. *Petrol. Tech.*, November 1940, A.I.M.M.E. Tech. Pub. No. 1259, 1–6.—The operator of every gas-recycling project is faced with two problems: (*a*) To remove a maximum quantity of the desirable hydrocarbons from the reservoir fluid, and (*b*) to return the residual gas to the producing formation with the least possible expenditure of compressor horse-power. Some projects have given excellent recoveries whilst operating at compression ratios within the limits of good practice, but the continued discovery of gas condensate production at pressures of 3000–6000 lb./in.^{2} calls for improvements on the present processes.

The low-temperature condensation process using chilling to 0° F. has given 90% efficiency when operating on gas of relatively low condensate content at a pressure of about 300 lb./in.^{2} greater than the observed maximum condensation pressure, but there are indications of a reversal of the isotherm positions at high pressures which would apparently render low-temperature condensation processes relatively ineffective at extremely high pressures. This is especially true for gases of low condensate content.

A commercial absorption plant working at 1200 lb./in.^{2} has been successfully operated with an efficiency of 98%. The absorption oil must be carefully controlled, and differs considerably from the conventional low-pressure absorption oils. Oil circulation rate is important.

A discussion of the paper by K. Eilerts is included. G. D. H.

126.* Methods of Interpreting the Findings in Core Analysis Work. F. Hornkohl. *Petrol. Eng.*, December 1940, **12** (3) 46.—After cleaning the outside of the core which is likely to be mud-contaminated, the degree of cementation, grain size and shape, and bedding are noted. The oil and water content is determined by distilling 100–150 gm. of the crushed sand. The minimum oil saturation so determined for such a sample to have commercial production at a certain depth is tabulated. The quartz lamp gives a marked fluorescence in the presence of light petroleums which might not colour extracting solvents, but not with mere gas sands. Connate water may be 30–70% of the total saturation, and is higher the lower the permeability of the sand. The sample for this measurement must be taken from the centre of a large core so that it will not be affected by drilling mud water, and the same is true for the salinity measurements

The permeability is measured along and perpendicular to the bedding, and the same specimen is used for the porosity determination. These two factors enable estimates of the storage capacity and the probable productivity of the reservoir rock to be made.

Grain-size analyses are useful for fixing the size of liner slots. The oil gravity in the reservoir can be estimated from the gravity of that distilled from the core.

G. D. H.

127.* Principles of Allocation in Oil Conservation. J. E. Pogue. *Oil Wkly*, 9.12.40, **100** (1), 12–15. Cf. *Oil Gas J.*, 12.12.40, **39** (31), 19.—The present system is outlined and explained and suggestions are made to modify it. The principles and procedures involved in the new system for conservation are :

Proration should be based on conservation, with equity and equilibrium as subordinate but essential supports. The central principle of conservation is the optimum rate of production, and this engineering criterion should gradually supersede economic criteria to the fullest extent possible in the techniques of allocating market demands. The maintenance of equity and equilibrium requires that changes in established practices shall be graduated transitions.

Forecasts of the total market demand by the U.S. Bureau of Mines should be founded on the broad base of total demand for all oils and recommendations made to the States by grades of crude. The allocations of total market demand to States, as now done on the trade channel procedure, is workable, but the optimum rate factor should be gradually accorded weight in the formula. State allowables should be broken down to regional quotas to serve as the basis for computing field allowables. Field allowables should be derived from the regional allowables and established on a basis gradually shifting to a common ratio to optimum rates.

The evolution away from the well as the unit of proration within the individual field should be encouraged in favour of the developed reserve as a function of the optimum rate. The developed reserve should be used in place of optimum rates for one or two years if necessary. A. H. N.

128.* Prevention of Flood Delays. Anon. *Oil Wkly*, 9.12.40, **100** (1), 16.—Methods to be followed in preventing delay of production by floods are described, and these consist of various simple and elaborate precautions, ranging from building earth mounds around the well to substantial reinforced-concrete constructions. Certain wells have been protected from heavy driftwood by erecting triangular cages of tubular steel around the Christmas tree, which cages will permit uninterrupted flow of water past the well, but will divert any heavy driftwood.

Temporary measures are also described in which use is made of sandbags, twig mattresses, and current chokes made of junk automobile bodies either weighted down by filling with earth or anchored by pipes to the soil. These measures are useful where floods usually take a few days before they assume serious proportions, and thus can be guarded against in time. A. H. N.

129.* Patents on Production. L. A. Layne. U.S.P. 2,223,374, 3.12.40. Appl. 30.9.38. Apparatus and method of gravelling wells, using two strainers of different meshes, one to exclude the oil sand but not the gravel, and the other to exclude both.

R. L. White and O. W. Lyons. U.S.P. 2,223,397, 3.12.40. Appl. 18.4.38. Treatment of wells with acid and using an aqueous dispersion of an organic jellifying material to protect permeable strata which are not to be acid-treated.

A. L. Cox. U.S.P. 2,223,602, 3.12.40. Appl. 4.10.38. Sand sucker apparatus for use in oil wells.

A. J. Penick and K. T. Penick. U.S.P. 2,223,702, 3.12.40. Appl. 28.3.38. Pressure control tubing-head with a valve controlled from outside.

H. Hügel. U.S.P. 2,223,786, 3.12.40. Appl. 13.1.38. Borehole pressure-gauge with a marking instrument mounted for rotary and reciprocating movements.

R. A. Gulick. U.S.P. 2,223,869, 3.12.40. Appl. 25.11.39. Pumping assembly.

J. L. Johnston. U.S.P. 2,223,913, 3.12.40. Appl. 4.9.37. Oil pressure tester with a timing mechanism to give a time-measured graph of pressure on a chart.

A. D. Garrison. U.S.P. 2,223,933, 3.12.40. Appl. 10.6.38. Treatment of oil wells to increase the proportion of oil relative to water produced by converting a water-wet producing sand to one which is preferentially wettable by oil, by depositing on the sand, without blocking, a film of water- and oil-insoluble metallic sulphide.

S. P. Hart. U.S.P. 2,223,936, 3.12.40. Appl. 16.2.40. Device for depositing chemicals of two different natures in a well simultaneously. A. H. N.

Transport and Storage.

130.* Methods of Studying Microbiological Anærobic Corrosion of Pipe-lines. R. F. Hadley. Part I. *Petrol. Engr*, March 1940, **11** (6), 171, and Part 2, *Petrol. Engr*, April 1940, **11** (7) 112.—In order to direct attention to the seriousness and widespread occurrence of corrosion by anærobic sulphate-reducing bacteria, a review is presented of the factors controlling this type of corrosion, methods of identification of its presence, and means of pipe protection against it. An examination of over 1500 test holes in pipe-lines in 16 counties in the eastern States revealed that from 6·7% to 72·9% of the test holes in each country were undergoing bacterial attack and that a high proportion of the pipe required protection against this corrosion. The HCl test for ferrous sulphide previously recommended for the detection of microbiological anærobic corrosion by the author (*Oil Gas J.*, 21.9.39, p. 92) has been criticized, but in every case where positive results have been given by it, or by laboratory analysis, cultures of sulphate-reducing bacteria have been grown from the location. It has also been proved that the sulphur present in the steel is insufficient to account for the sulphur found in the corrosion products. It is recommended that the ferrous sulphide test be checked by attempting to grow the bacteria (*Sporovibrio desulphuricans*) from its corrosion products, since the conditions under which bacterial corrosion and long-circuit differential æration corrosion proceed are identical and often found together. It is believed that the extent of pipe undergoing bacterial corrosion can be correlated with the degree of anærobiosis present. The factors affecting the development of microbiological corrosion include the degree of anærobiosis, the p_H of the soil water and the presence of a sulphur-oxygen salt. A number of organic compounds have been found to be available as hydrogen donators to certain strains of sulphate-reducing bacteria. Compounds formed in nature from sewage or cellulose, etc., decomposition or fermentation may give rise to conditions favourable for sulphate-reducing bacteria, so that the presence of wooden skids, tree trunks, vegetation, etc., in a pipe trench may give rise to corrosion by this means.

The p_H value is useful in determining the likelihood of serious bacterial corrosion, whilst SO_4 and CO_2 determinations have so far shown little correlation.

With regard to the life of pipe-line, it has been found that the rate of corrosion increases with time, probably owing to the fact that products of corrosion are maintained in the ferrous state and are not precipitated as impermeable membranes of ferric hydroxide. There is at present insufficient evidençe on the value of cathodic protection currents against bacterial corrosion. Use is being made of petroleum asphalt coatings encased in a box of durable wood. C. L. G.

Crude Oil.

131.* Removal of Inorganic Salts from Petroleum. C. M. Blair, Jr. *Refiner*, April 1940, **19** (4), 107–110.—The presence of inorganic salt in crude oil leads to two principal troubles—namely, stoppage of flow or decreased throughput, and corrosion. These, in turn, lead to accelerated coke formation, decreased heat transfer, increased replacement costs, lengthened shut-down time, increased labour requirements and lowered quality of products.

The presence of inorganic salts in crude oils almost invariably means that the oil contains emulsified brine—or, as it is frequently called, " BS." Examination of " BS " under the microscope reveals that it consists of minute droplets of a liquid dispersed in a continuous phase of oil. The dispersed liquid is water or an aqueous solution of salts. Cases have been reported where actual microscopic, cubical crystals of NaCl may be seen dispersed in the oil. The occurrence of solid salt dispersed in crude oil is, however, rare. Where solid salt is found in crude oil, it is often due to some treatment received by the oil after its production. Solid salts sometimes result from the evaporation of water from an emulsion which originally contained a concentrated brine. High-temperature field treating is often responsible for the occurrence of solid salt in oil.

Mechanical methods of desalting involve sedimentation or filtration and the paper briefly describes these. However, the chemical methods used to remove these inorganic constituents represent the major section of the paper.

In an average or typical plant, demulsifier is mixed with crude at the charge pump ; fresh water is introduced after the oil has been heated through the first exchanger. The water is dispersed in the oil by passing the mixture through a globe valve, giving a pressure drop of 10–15 lb. The mixture then passes through another exchanger and goes into a settling drum having a capacity sufficient to give 1½ hrs. settling time. Water is drawn continuously from this drum, and clean, desalted oil leaves at the top.

The chemical desalting method has definitely established itself as a successful commercial method. As shown by the examples given, the amount of demulsifier required varies with the type of oil, the salt content, and manner in which the reagent is used. It is emphasized that the use of a chemical reagent is not alone sufficient for successful removal of salt or brine from crude oil. It must be selected and employed in the proper manner, and its action must be complemented by the installation and operation of a properly designed system. A. H. N.

132. Aid in Interpreting Crude-Oil Analyses. H. M. Smith. U.S. Dept. of the Interior, Bur. Mines, Technical Paper 610, 1940.—The author of this paper suggests a means whereby analyses of crude oils may be extended to emphasize not only similarities between a number of samples, but equally important differences not usually directly apparent as a result of such analyses. Using boiling-point and specific-gravity values as a basis, an index number can be found, the magnitude of which indicates certain characteristics of a crude-oil distillation fraction.

Thus, a fraction composed exclusively of normal paraffin hydrocarbons would have zero as its index number, a fraction from a paraffin base crude oil of the usual type would have a small index-number, and fractions from intermediate naphthene-base crude oils increasingly high index-number values.

The actual equation which has been developed for calculating the correlation index of a fraction is as follows :—

$$\text{C.I.} = \frac{48640}{K} + 473{\cdot}7\,G - 456{\cdot}8$$

where C.I. is the correlation index, K the average boiling point of the fraction in degrees Kelvin (degrees Centrigrade plus 273), and G is the specific gravity of the fraction at 60° F./60° F.

Use of this correlation index should provide a useful alternative to the " base " system of classification of crude oils which is frequently ambiguous. For instance, naphthene-base oils, in addition to naphthenes may contain asphaltic material which is not naphthenic, aromatic hydrocarbons, and even paraffin wax. The correlation index does not have the effect of grouping together crude oils in accordance with their properties. It gives direct information concerning the distillable portion of a crude oil. In this way defects of the " base " system of classification are eliminated ; differences as well as similarities can be assessed, and there are no doubtful cases of classification.

H. B. M.

Gas.

133.* Elimination of Hydrate Troubles. E. G. Hammerschmidt. *Refiner*, June 1940, **19** (6), 215–218.—Nearly all freezing troubles in natural-gas pressure systems are caused partly or entirely by hydrocarbon hydrates, crystalline chemical compounds of methane, ethane, propane, or *iso*butane in which water is present in simple molecular proportions. The trouble may be permanently removed by lowering the dew point of the gas to the minimum temperature of the system when operating at maximum pressure, *i.e.*, by partial dehydration.

The magnitude of the lowering of dew point which may be effected by any aqueous hygroscopic solution is a function of the nature of the material and the contact temperature, whilst the residual water-vapour content of the gas is also a function of the total pressure of the systems. Charts and tables are presented detailing the characteristics of various dehumidifiers, and a detailed discussion illustrates their significance in typical plant operations.

Solid drying agents may be used for practically complete removal of water from the system. The relatively small quantities necessary for treatment of high-pressure gas makes possible an economical design of a dehydration plant using solid agents. Where complete dehydration is not necessary, a part of the gas may be completely dried, and then mixed with the wet gas by-passing the unit, thus increasing the capacity of the plant. The operating characteristics of a silica gel adsorbent plant are briefly given.

A. H. N.

134.* Modern Practices in the Dehydration of Natural Gas. T. S. Bacon. *Petrol. Engr*, Midyear 1940, **11** (10), 82.—Greater use of natural gas dehydration methods is due to the increasing use of higher operating pressures on transmission lines (up to 3000 lb. per sq. in.), to the availability of more exact information on the effects of pressure on hydrate formation, and to reduction in dehydration costs resulting from technical improvements in processes. The effect of an increase in temperature is to increase the temperature at which hydrates will form—*e.g.*, natural gas from the Texas Panhandle field is subject to freezing at 35° F. if the pressure is 125 lb. per sq. in., but at 52° F. if the pressure is 500 lb. per sq. in. Dehydration processes are of four main types : (1) Refrigeration to condense water vapour. (2) Absorption of water vapour in inorganic brines—*e.g.*, $CaCl_2$. (3) Absorption in liquid organic chemicals—*e.g.*, diethylene glycol. (4) Adsorption on solid materials—*e.g.*, activated alumina.

(1) Refrigeration causes partial dehydration if the dew point of the gas is above the temperature at which the gas leaves the coolers, but is limited by the formation of hydrates, causing plugging, if operation is below this temperature.

(2) Absorption in $CaCl_2$ solution is widely used, the solution being cheap, stable, and fairly effective, but tending to become corrosive and to solidify on cooling or concentration. The maximum dew-point depression obtainable is less than 30° F., actual figures being 20–25° F. Improved results are given by combining the plants with refrigeration units.

(3) Absorption in glycerine is seldom used owing to its cost and instability, although giving a greater dew-point reduction than $CaCl_2$ solution. Diethylene glycol gives a dew-point depression of 45° F., is non-corrosive, and does not solidify. It was used in 16 plants in 1939, and is useful in combination with amines for sour gases.

(4) Solid adsorbents—activated alumina and silica gel—can dehydrate gas almost completely, the former being in commercial use. Anhydrous calcium sulphate and solid calcium chloride are also used as absorbents.

Type 1 plants are the cheapest to operate, type 3 being slighty dearer than type 2, although causing less trouble and being more efficient. Operating costs on types 2 and 3 plants vary from 7 to 75 cents per million cu. ft. of gas. Selection of the most suitable type of plant must take into account the dew-point depression obtained and plant location (*i.e.*, using a single large plant or a number of smaller plants). In view of the high pressures in field lines, there is a considerable field of application for small self-contained fully automatic dehydration plants for well-head use.

C. L. G.

Cracking.

135.* Houdry Catalytic Process. O. W. Wilcox. *World Petrol.*, October 1939, **10** (11), 51.—The Houdry process is based on three groups of interrelated inventions viz.: (1) the catalyst; (2) the catalyst cases for contacting the feed-stock with the catalyst; (3) the methods by which the reactions are controlled.

The catalysts consist of natural or prepared clays, the effectiveness of which may be supplemented or modified by the addition of metals or metallic oxides. The catalysts are prepared in granular form, the treatment being controlled to give the required activity.

Regeneration is effected by burning off deposited carbon in a stream of air, and a feature of the catalysts is freedom from loss of activity when subjected to the heat treatment necessary for regeneration. Catalyst masses are stated to have withstood thousands of regenerations without serious depreciation.

The catalyst cases have to withstand severe conditions, in that there is frequent alternation between on-steam operation at about 840° F. and catalyst regeneration at about 950° F. Heat transference is accomplished by a circulating system in the cases, and the whole apparatus is designed to effect exchange of heat as rapidly as possible.

A considerable number of variables in the process—*e.g.*, pressure, temperature, time of reaction, etc.—are under accurate control. This makes the process flexible, in that the products from a given raw material may be controlled to meet specific requirements. The adaptability of the process has made possible the transformation of residues.

Notable results of Houdry cracking are: (1) high yield of gasoline; 44–46% of charge; (2) high octane rating of the gasoline; and (3) high recovery; in many cases the volume of gasoline and gas oil produce is almost equal to the charge. The quality of the products is claimed to represent a distinct advance over thermally cracked products.

Data on a Houdry plant processing 13,000 brl. per day show an operating cost of 6–8 c. per brl., as compared with 12–14 c. estimated for thermal cracking. Moreover, a greater appreciation in the value of the products is claimed for Houdry cracking as compared with thermal cracking.

Comprehensive data on the results of catalytic cracking operations are included, and a detailed description of a Houdry plant operating at Marcus Hook is given.

R. J. E.

136.* Era of Catalytic Cracking. M. B. Cooke. *World Petrol.*, October 1939, **10** (11), 72.—The development of catalytic cracking is associated with the increasing demand for high-octane aviation fuels. The development in recent years of high-octane gasoline producing plants is reviewed. The processes reviewed include :—

1. Re-forming of naphtha. The relative merits of reforming cum polymerization and naphtha reversion are discussed.
2. Polymerization of unsaturated gases and pyrolysis of saturated gases.
3. Production of *iso*-octanes by polymerization and hydrogenation and by alkylation.
4. Production of aromatics from petroleum gases by pyrolysis.
5. Catalytic cracking and catalytic reforming.

The merits of the various processes from a consideration of the raw material used and its availability, and the yield and quality of the products are reviewed.
R. J. E.

137. Patent on Cracking. M. Pier and W. Simon. U.S.P. 2,224,003, 3.12.40. Appl. 24.3.39. An initially highly active catalyst, the activity of which has been reduced by its employment in a pressure hydrogenation treatment of carbonaceous substances poor in hydrogen, is used in the cracking of hydrocarbons containing more than 15 gm. of hydrogen per 100 gm. of carbon.
H. B. M.

Polymerization.

138.* Thermal Reforming Plus Catalytic Polymerization. R. P. Mase and N. C. Turner. *Refiner*, April 1940, **19** (4), 111–112.—By combining thermal reforming with catalytic polymerization, a definite improvement is made, first, in reduction of losses, and, second, in increase in octane number, as compared to the results of straight reforming. From a plot of experimental data it is apparent that thermal reforming alone will produce a 70-motor-method octane product with an 80% yield of product, based on feed volume, whilst with catalytic polymerization added, a 70-motor-method octane can be produced with 85% yield of product. In short, on an equivalent octane basis, a 5% increase in yield, on an equivalent 70 octane is had by adding polymerization, or, conversely, the loss is cut by 5%. On the basis of an equivalent yield of, say, 85%, the octane may be increased by 2·5 points, with the addition of polymerization.
A. H. N.

139.* Induced Pyrolysis of Methane. W. J. Hessels, D. W. van Krevelen, and H. I. Waterman. *Refiner*, April 1940, **19** (4), 127–130.—In the pyrolysis of methane the primary change is the formation of radicals which react with each other and with the undecomposed methane molecules.

The same radicals can also be formed by the chemical action of, *e.g.*, halogenes on methane or by the thermal decomposition of aliphatic hydrocarbon groups which are less stable than methane itself. Hydrocarbons, especially aliphatic hydrocarbons, such as propane, butane, etc., and hydrocarbon derivatives such as halogen and sulphur compounds and oxygen compounds, which are able to split off hydrocarbon radicals, can be used for the production of the radicals necessary to demonstrate the induction effect on the methane pyrolysis.

The inductor effect has been shown without any ambiguity by experiments with pure methane under comparable conditions, and also by experiments with the inductor in a pure state in a nitrogen medium.

By applying the inductor effect of foreign substances it has been shown that methane can be transformed into unsaturated hydrocarbons (ethylene, acetylene, benzene, naphthalene) under such conditions that methane alone passes the heating zone unchanged.

The ratio of methane to inductor in the ingoing gas mixture is of importance. The inductor effect is more marked if the concentration of the inductor molecules is smaller compared with the methane concentration.

Aliphatic molecule structures are essential for the inductor action ; aromatic compounds such as benzene and naphthalene had very little or no induction effect on the methane pyrolysis in the authors' experiments.

High-percentage conversions of methane are observed when the gases are passed through the furnace several times. Unsaturated aliphatic gases formed in the first passage through the heated tube act as inductor in the next passage.

Induced pyrolysis can be applied to methane present in natural gases. These gases contain, in addition to methane, substances capable of acting as inductors.

It has been shown that by the decomposition of the higher hydrocarbons present in natural gas, methane can partly be involved in the decomposition under conditions in which pure methane undergoes no decomposition.
A. H. N.

140.* Gulf Polyform Process. P. Ostergaard and E. R. Smoley. *Refiner*, September 1940, **19** (9), 301–312.—The Polyform process differs from the ordinary thermal cracking process in that the oil is processed in admixture with varying amounts of normally gaseous hydrocarbons, particularly propanes and butanes, and in that the oil is subjected to higher temperatures and higher degrees of conversion/pass than would be possible, under otherwise similar conditions, without excessive coke formation in the cracking tubes. The process results in higher yields of gasoline of higher octane rating than can be obtained by subjecting the same oil to conventional thermal cracking operation.

In discussing the theory of the process in some detail, it is pointed out that any process by which crack/pass can be increased means higher octanes and greater yields of light products. The gasoline yield can be further increased by converting part of the gases produced into gasoline hydrocarbons. The gaseous hydrocarbons are introduced into the oil in the coils in order to effect a higher degree of conversion of the oil when diluted with C_3 and C_4 hydrocarbons than is possible without the use of C_3 and C_4 gases; and, further, to effect side reactions and polymerizations with the C_3 and C_4 themselves. The temperature of the mixture must be above the critical temperature so that a solitary homogeneous gaseous phase results in the coil, thus obviating the settling of a liquid film on the coil with many resultant bad effects on the yield and quality of the products. This subject is discussed in detail.

Flowsheets and descriptions of typical Polyform plants are given with data on yields and qualities of products obtained from various crudes.

The octane number of Polyform gasoline depends, like the numbers of all other cracked gasolines, on the characteristics of the charging stocks. Naphthenic stocks, in general, yield gasolines of the highest C.F.R.–A.S.T.M. octane numbers obtainable by this process, whilst the paraffinic stocks yield the lowest numbers. However, in Polyform cracking, it is always possible to produce gasoline with an octane number of 75 to 77, regardless of the characteristics of the charging stock. Illustrations of these facts are given.

Polyform gasoline is characterized by having a very much higher appreciation between C.F.R.–A.S.T.M. octane ratings and C.F.R.-Research octane ratings than ordinary thermally-cracked gasoline. Treating Polyform gasoline for motor and aviation petrol production is discussed. A. H. N.

141. Some New Developments in the Petroleum Industry. W. D. Williams. *Chem. and Ind.*, 1941, **60**, 22–24.—The outstanding developments in the petroleum industry over the last few years have been due to the efforts to increase the octane numbers of motor spirit. Between 1931 and 1939 average octane numbers of first-grade motor spirit rose from 60 to 74. The various steps to obtain the higher-performance fuels are reviewed.

Thermal and catalytic polymerization of refinery gases are capable of very great potential development. It is estimated that 3000 billion cub. ft. of the necessary gases are produced annually and that such a quantity could be converted to 1500 million gallons of motor spirit.

The catalytic polymerization is much more productive than the thermal process—*e.g.*, average yields of the former are 8·3 gal. per 1000 cub. ft. and of the latter 3·7 gal. per 1000 cub. ft.

The Houdry catalytic cracking process has been developed to avoid the more drastic thermal cracking. The process is said to convert 85% of the charging stock to motor spirit with an octane number of 78–80.

The developments of polymerization and alkylation have resulted in, for instance, *iso*-octane now being produced in vast quantities at one hundredth its cost in 1930 (when it was £6 per gallon). Further, these developments have led to plants being contemplated for the manufacture of 150 Octane No. fuels.

The use of refinery gas as a raw material for the manufacture of a wide series of chemicals is reviewed. These chemicals include many alcohols, ketones, ethers, and ethylene glycol. The production of cheap acetylene by submitting refinery gas to the electric arc has led to the development of the chlorinated acetylene group of solvents, of neoprene and the vinyl resin group of plastics.

Short notes appear on the solvent refining of lube. oils and on the production of synthetic rubbers. T. C. G. T.

142.* Applications of Thermal Polymerization. Humble Oil & Refining Co., Phillips Petroleum Co., and the M. W. Kellogg Co. *Oil Gas J.*, 22.6.39, **38** (6), 52. *Paper presented before 1939 annual meeting N.G.A.A., Tulsa.*—A comprehensive discussion is given of the design, operation, and economics of thermal polymerization plants based on the results obtained from two plants in operation. Five different flow-sheets are compared and analysed and the effect of various plant modifications discussed. The relative values of paraffin hydrocarbons as polymerization feed-stocks are compared, and a suggested design given for thermal polymerization, to be used on natural gasoline plants.

The two plants described are : (*a*) a polymerization unit at a Baytown Tex. refinery of capacity 8000 bbl. per day liquid butane with a complete propane-recovery system, producing light and heavy polymer gasoline streams, and (*b*) a smaller unit at a Borger Tex. refinery of capacity 2500 bbl. per day, in which complete propane recovery is not attempted, and producing one polymer stream which is vapour-phase clay treated. For commercial operation on paraffin hydrocarbons, *n*-butane is the most valuable, *iso*butane next, and propane third, ethane being of little value. Under the same conditions of pressure, but increasing the crack per pass, the ultimate yield of polymer decreases, and does not allow of long runs. On the other hand, with a smaller crack per pass, a larger-sized furnace, stabilizing unit and recovery system are required. In general, quenching is resorted to, where possible, in order to retard coke deposition and increase the length of run, so that polymerization units operating in connection with refineries (having quenching oils available) are designed to operate at a high crack per pass, whilst those in connection with casing-head plants are based on a low crack per pass. For processing propane a furnace soaking volume 3·83 times as great as that for butane is required. The economics of the two plants in question are finally discussed, direct comparison being difficult, owing to different methods of accounting and basic values. C. L. G.

143. Patents on Polymerization and Alkylation. Texaco Development Corp. E.P. 527,720, 15.10.40. Appl. 13.4.39. Alkylation of hydrocarbons by subjecting olefinic or olefine-containing hydrocarbons and *iso*paraffins or *iso*paraffin-containing hydrocarbons to the action of strong sulphuric acid in a reaction zone under alkylating conditions. In this way the *iso*paraffins are alkylated by the olefines to form saturated branched-chain hydrocarbons. Thereafter the reaction mixture is withdrawn and a portion thereof recyclized to maintain a higher ratio of acid and *iso*paraffins than was present in the initial mixture.

I.G. Farbenindustrie A.-G. E.P. 527,767, 16.10.40. Appl. 14.3.39. Process for the isomerization of paraffinic hydrocarbons having from four to twelve carbon atoms in the molecule. The hydrocarbons are passed at a temperature between 200° and 600° C. and at a pressure above 100 atmospheres, together with hydrogen having a partial pressure of less than 5% of the total pressure, over catalysts having an adsorbent or hydrogenating or dehydrogenating action.

N.V. De Bataafsche Petroleum Maatschappij. E.P. 527,805, 16.10.40. Appl. 20.4.39. Method of reducing the cost of polymerizing olefines by prolonging the life of polymerization catalysts. This is effected by washing the olefines with water prior to contacting them with the catalyst. The invention has particular reference to phosphoric acid catalysts, but is equally applicable to other phosphorus-containing catalysts, sulphuric acid polymerization catalysts, or non-acid catalysts known to be capable of polymerizing olefines.

Anglo-Iranian Oil Co., Ltd. E.P. 528,178, 24.10.40. Appl. 16.3.39. Use of anhydrous ferric chloride as a catalysis-promoter in the process of isomerization of normal paraffins.

Texaco Development Corp. E.P. 529,194, 15.11.40. Appl. 24.5.39. Alkylation of *iso*paraffins with olefines by treating the olefines and an excess of low-boiling *iso*paraffins with concentrated sulphuric acid under alkylating conditions. Thereafter the used acid is separated from the hydrocarbon reaction products and contacted separately with olefine-free *iso*paraffins to strip it of absorbed hydrocarbons.

Texaco Development Corp. E.P. 529,197, 15.11.40. Appl. 24.5.40. Alkylation of low-boiling *iso*paraffins with olefines by charging *iso*paraffins, olefines, and liquid acid catalyst to a reaction zone at such a rate that a predetermined acid to hydrocarbon ratio is maintained in the fresh feeds to the zone. The acid and hydrocarbons are contacted in the zone to obtain alkylation of the *iso*paraffins by the olefines, and the hydrocarbon reaction products and acid thereafter withdrawn. Subsequently the acid and hydrocarbons are separated and the acid recyclized to the reaction zone in an amount sufficient to maintain a higher ratio of acid to hydrocarbons in the reaction zone than in the fresh feeds.

Texaco Development Corp. E.P. 530,250, 9.12.40. Appl. 31.5.39. Manufacture of motor fuels and production of hydrocarbons of gasoline boiling-point range by the alkylation of *iso*paraffins, such as *iso*butane, with normally gaseous olefines.

V. N. Ipatieff and A. V. Grosse. U.S.P. 2,217,019, 8.10.40. Appl. 28.2.38. Production of hydrocarbons boiling within the gasoline range by reacting *iso*butane with a normally gaseous olefin in the presence of boron fluoride, nickel, and hydrogen fluoride.

F. R. Moser. U.S.P. 2,218,135, 15.10.40. Appl. 16.5.34. Preparation of a liquid motor fuel having incorporated therein small quantities of methyl ethyl ketone peroxide.

C. A. Kraus. U.S.P. 2,220,930, 12.11.40. Appl. 3.11.38. Polymerization of hydrocarbons of the olefin series by contacting them with a compound of the formula $MXmRn$, where M represents a member of the class consisting of aluminium, gallium and boron, X represents a halogen, R a monovalent hydrocarbon radical, and m and n each represent an integer from 1 to 2, inclusive, $m + n = 3$.

W. E. Kuentzel. U.S.P. 2,221,000, 12.11.40. Appl. 11.8.37. Improvement in the process of polymerizing hydrocarbons containing *iso*butylene into resins in the presence of a catalyst.

A. Wassermann. U.S.P. 2,224,071, 3.12.40. Appl. 18.5.38. Treatment of olefin hydrocarbons with a combined catalyst consisting of a heavy metal sulphide and a heavy metal sulphate. The olefins and the catalyst are subjected to a temperature between 100° and 200° C. until a part of the olefins is converted into polymers the boiling point of which is at least 50° C. higher at atmospheric pressure than that of the original material.

M. M. Holm, E. H. Oakley, and R. L. Humphreys. U.S.P. 2,224,102, 3.12.40. Appl. 26.6.39. Alkylation of *iso*paraffins with olefins by contacting *iso*paraffin and olefin with sulphuric acid of alkylating concentration and maintaining the concentration of alkyl acid sulphate in the acid below about 1% by weight of the acid.
H. B. M.

Synthetic Products.

144. Synthesis of Petroleum-like Hydrocarbons from Carbonaceous Material. R. Fussteig. *Petrol. Engr*, May 1940, **11** (8), 95.—The development of water-gas manufacture as a source of gasoline is reviewed. Water-gas can be manufactured from a variety of solid and gaseous fuels : coke, various coals, wood, peat, natural gas, and coke-oven gas. In each case the composition of the water-gas depends on the operating conditions, a temperature of 1100–1200° C. being considered the most suitable to give a CO : H ratio of 2 : 1. The use of catalysts prevents the formation of carbon and promotes the decomposition of methane. A detailed description and flow-sheet of the manufacture of water-gas from coal are given. Steam is injected into the reaction chamber at the moment when the coal decomposes into gases and coke. Reaction of coke with steam and transformation of gases into water-gas occur simultaneously. Before the water-gas can be used for gasoline synthesis, removal of hydrogen sulphide and organic sulphur compounds is essential, to avoid poisoning of the catalyst subsequently used. The former is usually effectively removed by iron oxide, whilst iron oxide plus alkali carbonate and this mixture in combination with cobalt sulphide are used for the removal of sulphur compounds.

The formation of gasoline from water-gas is attributed to a first reaction into $\equiv CH_2$ molecules which are then subject to polymerization, isomerization, and alkylation

reactions. The hydrocarbons produced consist largely of straight-chain paraffins, but changes of the operating conditions have a considerable effect on the product. Conditions under which paraffin wax is produced from water-gas are described.
R. J. E.

145. Patents on Synthetic Products. N.V. De Bataafsche Petroleum Maatschippij. E.P. 527,954, 18.10.40. Appl. 24.4.39. Preparation of saturated hydrocarbons with branched or more highly branched chains from hydrocarbons boiling below about 160° C. and having five or more carbon atoms in the molecule.

M. W. Kellogg Co. E.P. 527,975, 21.10.40. Appl. 23.4.38. Method of increasing the aromatic content of a mixture of hydrocarbons containing *cyclo*-olefins, naphthenes, and aromatic hydrocarbons. The mixture is subjected to dehydrogenation in order to convert the *cyclo*-olefins into aromatic hydrocarbons and afterwards to mild conditions of hydrogenation to convert any residual olefins.

A. V. Grosse and W. J. Mattox. U.S.P. 2,217,010, 8.10.40. Appl. 31.5.38. Production of aromatic hydrocarbons from acetylene hydrocarbons by dehydrogenating and cyclicizing the acetylene hydrocarbon by subjection to a temperature between 450° and 700° C. for 0·1 to 30 sec. in the presence of a compound selected from the class consisting of vanadium, columbium, and tantalum.

O. W. Cass. U.S.P. 2,218,018, 15.10.40. Appl. 2.5.38. Manufacture of 1 : 4-dihalogen butane by reacting tetramethylene oxide with a hydrohalogen acid in the presence of a dehydrating acid.

W. Friedrichsen and W. Fitzky. U.S.P. 2,218,640, 22.10.40. Appl. 18.11.39. Production of aliphatic hydrocarbons containing conjugated double linkages by contacting a 1 : 3-dioxane with a solid dehydration catalyst. H. B. M.

Refining and Refinery Plant.

146. Improvement in Octane Number and Lead Susceptibility. D. L. Yabroff and A. C. Nixon. *Oil Gas J.*, 22.2.40, **38** (41), 74.—The effect of sweetening with " Solutizer " solution (potassium hydroxide and potassium *iso*butyrate) on octane number and lead susceptibility is described. In contrast to the conventional sweetening processes which convert mercaptans into disulphides, the " Solutizer " process extracts the mercaptans from sour gasoline. Data obtained on a large number of different types of gasolines before and after sweetening with " Solutizer " show that significant improvement of both octane number and lead susceptibility is achieved by this sweetening process. It is held that as disulphides are somewhat more detrimental to lead susceptibility than mercaptans, the saving of T.E.L. when using the " Solutizer " process as compared with that when using the conventional sweetening processes is more striking than the data provided indicate. R. J. E.

147.* Cold Acid Treatment of Cracked Distillate. Anon. *World Petrol.*, October 1939, **10** (11), 76.—A detailed description of a Stratcold plant for the cold acid treatment of heavy cracked distillate to remove sulphur compounds is given. The process consists essentially of contacting the raw distillate with 98% sulphuric acid under precisely controlled temperature conditions in a three-stage, counter-current treating system. The treated distillate is passed through a clarifier, a two-stage water-wash system, and finally contacted with caustic soda for neutralization. The distillate is treated with 9 lb. sulphuric acid per barrel, and the treating temperatures at the three stages are maintained at 30° F., 30° F., and 20° F. respectively. The equipment and method of control are described in detail. R. J. E.

148.* World's Refining Facilities. Anon. *World Petrol.*, October 1939, **10** (11), 86.—Details are given of world refining facilities (excluding U.S.A.). Plants are classified by continents and by countries, and their locations and capacities given. The position of belligerent countries in relation to their requirements of petroleum products is discussed. The development and installation of plants operating recently developed processes—*e.g.* catalytic cracking, polymerization and alkylation, are reviewed. R. J. E.

149.* Alkylation's Place in Refining Industry. Anon. *World Petrol.*, October 1939, **10** (11), 104.—Growing demand for high-octane aviation fuel has directed particular attention to alkylation as a simpler method for the production of *iso*-octanes. Alkylation consists of reacting *iso*butane with butylenes in the presence of 98% sulphuric acid at 45–60° F. The reactants are mixed in the ratio of 1·2 parts *iso*butane with 1 part butylene. The raw alkylate is separated from the acid by gravity and fractionated to yield 92% of *iso*-octanes with an endpoint of 310° F., 2–4% of a heavier fraction with an endpoint of 400° F., which is suitable for motor-fuel blending, and a residue having an endpoint of 600° F., which can be used for power kerosine or diesel oil.

Alkylation has a distinct advantage over polymerization for the production of *iso*-octanes, in that *iso*butane is used, and so the yield is roughly twice that obtained when using refinery gases. Moreover, the hydrogenation step is eliminated in alkylation. However, whilst *iso*butane is plentiful in natural gasoline, it cannot always be delivered cheaply at the refineries. R. J. E.

150.* Catalysis in Petroleum Refining. Anon. *World Petrol.*, October 1939, **10** (11), 62.—The rôle of catalysts in petroleum refining is reviewed. The catalytic processes used in the industry fall into three groups :

(1) Break up of high molecular weight hydrocarbons (catalytic cracking).
(2) Change of product to a more desirable form (catalytic reforming).
(3) Combination of hydrocarbons to form a synthetic product of greater value (catalytic alkylation, polymerization, and hydrogenation).

Catalytic cracking represents an improvement on thermal cracking in that superior products are produced at lower temperatures and pressures. The better-quality gasoline yielded in catalytic cracking is attributed to the formation of cyclic and branch-chain hydrocarbons superior in quality to the straight-chain hydrocarbons which preponderate in thermally cracked gasoline.

In catalytic reforming the products of thermal or catalytic cracking can be improved by passage over a catalyst, and results show that this has a distinct advantage in quantity and quality over thermal reforming. The development of catalytic reforming from clay-treating processes is reviewed and present-day catalytic reforming processes are outlined.

In catalytic polymerization the C_3 and C_4 hydrocarbons form the main raw material. Various processes for the catalytic polymerization of these lower hydrocarbons to high-quality gasoline are described. Details are also given of the processes for the polymerization-cum-hydrogenation of butylenes to *iso*-octane, the combination of butylenes with *iso*butane to give alkylate octanes, and of ethylene with *iso*butane to give neohexane. R. J. E.

151.* Equipment Inspection for Greater Refinery Safety. P. N. Gammelgard. *Refiner*, January 1940, **19** (1), 8–15.—Causes contributing to failure of cracking units are classed under two types. The first is the human element, and is briefly discussed. The effect of temperature in reducing the strength of metals is not generally well understood, and many who would refuse to increase the operating pressure on a tower by 50 lb./sq. in. would not hesitate to increase the temperature 50° or 100° F., although the latter procedure might, in certain cases, be hazardous.

The fact that many parts of a cracking unit operate at temperatures above the boiling point of water points out the potential danger of introducing water in any appreciable amount into the system. The tremendous increase in volume which takes place when water flashes into steam may quickly build up pressures which relief valves will be unable to accommodate.

Modern cracking and combination units cannot be made foolproof. Regardless of the thoroughness of inspection, cracking units must be operated by careful, well-trained, and " safety-conscious " personnel.

The second class of causes contributing to equipment failure is composed of factors which to a large degree are beyond the immediate control of the operating personnel.

The adverse influence of these factors on the safety of equipment makes inspection a necessity.

Listed, without attempting to place them in the order of relative importance, they are : (1) corrosion, (2) erosion, (3) oxidation, (4) creep, (5) fatigue failure.

Refineries are fertile fields for corrosion in all its varieties. Examples of direct chemical action, pitting, dezincification, galvanic action, concentration-cell corrosion, corrosion cracking, and corrosion fatigue are common. Illustrations are described in detail to explain various methods and aspects of corrosion.

Erosion in refinery operations may be caused by any one of several factors or by a combination of them. Erosion may result from (1) high vapour or liquid velocities; (2) abrasive or gritty matter carried in the oil or vapour stream; (3) the junction of two streams; (4) coke-cleaning equipment; (5) friction between two objects. These items are briefly explained.

Flame impingement is a well-known cause of oxidation, although it is by no means a requisite for oxidation. Local overheating as a result of coke conditions or faulty firing may result in a badly oxidized tube within a short period. Under moderate oxidizing conditions, scale will form on the surface of the tubes. The insulating effect of the scale will in turn necessitate higher muffle temperatures in order to maintain the oil line, and if the situation is permitted to become progressively worse, a bulged tube will result, and ultimately a rupture.

Creep and fatigue are problems to be solved by the designers, although the operator has to understand their implications.

The determination of the inspection and testing periods should be the responsibility of the inspector in charge. The periods so determined will be the result of past findings, severity of operating conditions, and potential danger in the event of failure. Until an experience record has been built up, it is certainly better practice to " over-inspect " than " under-inspect."

A typical inspection procedure covers the following points : (1) visual examination; (2) drilling and calipering; (3) hammer testing; (4) pressure tests; (5) records and reports. These steps are detailed. A. H. N.

152.* Application of *X*-Ray Inspection to Oil Refining Equipment. H. R. Isenburger. *Refiner*, January 1940, **19** (1), 22–29.—By far the majority of large objects radiographed up to the present have been castings. Whether justified or not, there is a tendency on the part of designing and operating engineers to place confidence in forgings without subjecting them to *X*-ray inspection. On the other hand, they prefer castings for many services in oil-refinery installations, because castings may be cheaper even when they are *X*-rayed and the additional cost of the radiographic inspection is taken into account. Inspectors are strengthened in this tendency when assured of the quality of the castings. The degree of this assurance depends on their faith in the manufacturer and his workmen, and on tests applied to castings. The usual physical test consists of applying hydrostatic pressure to a value well in excess of the maximum intended working pressure. If the casting does not fail at this time, and if there is no visible evidence of damage as a result of the test, the casting will be accepted with the assumption that it has no serious defects and that it will stand the working pressure during a reasonable operating life.

This is shown not to be always a safe conclusion. An ell fitting was subjected to a hydrostatic test and apparently withstood it successfully. However, subsequent *X*-ray examination disclosed that the test had produced or extended a crack which had started from a small gas-hole or an inclusion of a foreign substance, and which had progressed about two-thirds of the way through the outer portion of the wall of the fitting. It is possible that because of this defect the ell would have failed while in service, and serious damage and injury to personnel and equipment would have resulted.

Various examples of inspecting welds and castings are studied and test technique is outlined. A warning is given against inexpert advice and interpretation of test results. A. H. N.

153.* Field Lining of Refinery Vessels with Stainless Steel Sheets or Strips. K. E. Luger. *Refiner*, February 1940, **19** (2), 41–46.—Field lining of existing vessels with stainless steel is now being done on a broad scale. Towers lined in the field nine or more years ago are still giving entirely satisfactory service, and indicate that there is no appreciable amount of corrosion even when handling sour crudes. While it is not claimed that a lining job can be done in the field as cheaply or as perfectly as in a fabricating shop while the vessel is being constructed, it nevertheless appears to be well established that field lining can be done both satisfactorily and economically. It is estimated that in the last 2½ years alone well over 150 vessels have been field lined with thin stainless steel. They range from small heat-exchanger shells to large vacuum towers, and include a goodly number of riveted towers.

These field liners of stainless steel have been applied in various ways; in fact, it is likely that no two refineries do the job in exactly the same way. The important considerations are the fastening of the liner to the steel securely at frequent intervals and sealing it so as to prevent entry of oil or vapour between the shell and the vessel. All successful jobs embody these two features and differ only in details. These details are given with illustrations and warnings against common faults.

A. H. N.

154.* Continuous Coal Distillation Similar to Petroleum Refining Technique. B. O. Lisle. *Refiner*, March 1940, **19** (3), 63–67.—Britain's leading manufacturer of synthetic-oil products from coal recently completed a new continuous-distillation plant which is the first of its kind to be based on the technique used in refining crude petroleum, as well as embodying the latest practice in coal-tar treatment. Better fractionation and more efficient washing have improved the quality of the products. The distillation unit is of flexible design and operation, enabling it to meet varying demands, but the usual yield is about 10% motor fuel, 15% diesel oil, 16–18% cresol, 59–61% creosote and fuel oil, and the remaining 20% pitch.

Great Britain ranks third in the world consumption of petroleum, requiring approximately 11,430,000 brl. of gasoline, 1,280,000 brl. of diesel oil, and 14,280,000 brl. of lubricating oil. Of this, however, only about 8% of total requirements are produced from indigenous sources; some from distillation of the hydrocarbon content of the Scottish shales, but most is from heat treatment of coal. Petroleum production is negligible, despite the several million dollars spent during the past five or six years in drilling operations all over the United Kingdom.

The plant is briefly described, with a tribute to Colonel Bristow's enterprising work.

A. H. N.

155.* Treatment of High-Sulphur Cracked Distillate with Stratcold Process. F. A. Apgar and C. A. Day, Jr. *Refiner*, April 1940, **19** (4), 97–101.—In the refining procedure, cracked distillate is split into a heavy and light fraction by the usual debutanizing and stabilizing operations. The heavy fraction, having a boiling range of 220–410° F., contains the refractory sulphur compounds and is subjected to the Stratcold treatment; the light fraction contains sulphur compounds which are satisfactorily removed by caustic washing. The acid-treated distillate is re-run and the overhead blended with the caustic-washed light fraction for sweetening.

In the Stratcold treatment, cracked distillate is contacted in three stages with counter-current flow of acid. The treated distillate is then passed upward through a clarifier or rock-packed tower for further separation of acid sludge, thence water-washed, and finally neutralized with caustic soda solution.

The plant was designed to treat 11,000 brl./day of heavy cracked naphtha with 9 lb. of 98% sulphuric acid/barrel of naphtha with temperatures on the stages maintained at 30° F., 30° F., and 20° F., respectively.

The plant and various equipment and instruments are described and data are tabulated for a test run. Operations are smooth and appear to be economical.

A. H. N.

156.* Solid Copper Sweetening Process. Anon. *Refiner*, April 1940, **19** (4), 113–116. —The reagent of the solid copper sweetening process is composed of fuller's earth impregnated with an aqueous solution of cupric chloride. The function of the fuller's earth is only that of a carrier for the cupric chloride solution, and in this form has no value as a treating agent.

Two separate engineering lay-outs are followed; one when the commodity contains both hydrogen sulphide and mercaptan sulphur, and the other when the gasoline is free of H_2S, and contains only mercaptans. To sweeten any gasoline, whether straight-run or natural, the material must be free from hydrogen sulphide before it enters the solid copper reagent treating towers. For the elimination of this sulphur compound, a series of vessels and operating equipment are included which utilize an aqueous solution of commercial caustic.

The plant's requirements and operations are detailed. Maintenance of a solid copper-sweetening plant is almost negligible, consisting almost entirely of maintaining the proper ratio of air to gasoline, and preventing the intrusion of excess suspended water or slugs of water from any source.

The total quantity of gasoline that may be sweetened with a given volume of copper chloride depends on the sourness of the gasoline, the absence of free water in the product as it enters the columns, the complete elimination of H_2S from the commodity before it enters the columns, and the common everyday attention any process should have. A. H. N.

157.* Developments in Distillate Recovery. L. S. Reid and R. L. Huntington. *Refiner*, May 1940, **19** (5), 131–137.—The development and recovery methods in distillate fields of the Gulf Coast are described. Retrograde condensation and distillation are briefly reviewed; interest in this field of scientific research has increased since industries began using extremely high-pressure techniques.

Repressuring and pressure maintenance in general is similarly reviewed and the importance of gas conservation, maintenance of a single gaseous phase in distillate reservoirs instead of a two-component system, the reduction of gas mixing between virgin distillate fluid and repressuring gas, and prevention of a too-rapid edge-water encroachment is stressed and explained.

In determining the optimum recycling pressures for plant design, many variables are to be considered: permeability, thickness and extent of the reservoir formation, the composition of reservoir fluid, reservoir pressure, and the relative value of distillate to that of dry gas are major items. However, the problem may be simplified by assuming constancy of these factors and solving by balancing recompression cost and distillate return both as functions of the pressure of the first-stage separator.

Few phases of the industry have seen such rapid improvements as distillate-recovery plants, and it is stated that certain projects became obsolete within a year from completion. The more important processes are described and illustrated. A. H. N.

158.* Butane Chilling of Absorption Oil Increases Plant Yield. Anon. *Refiner*, May 1940, **19** (5), 144–145.—By reducing the temperature of absorption oil by 20° F. an increase of 2000 gal./day of natural gasoline was obtained—not including improved yield of butane and lighter fractions. The temperature reduction is accomplished by expanding a normal butane cut in a closed system of shell-and-tube exchanger, through which butane is continuously circulated. The unit is compact and semi-automatic. To obtain the full benefit of butane cooling, the tubes through which the oil flows are about four-fifths submerged with butane liquid, following closely the flooded refrigeration systems used in refineries when dewaxing oils.

The quantity of oil cooled is 240,000 gals./day of 37–38 A.P.I. gravity, and the quantity of butane required to cool it 20° F. is 600,000 cu. ft./day. A. H. N.

159.* Synchronized Controls Improve Boiler Operation. Anon. *Refiner*, May 1940, **19** (5), 151–152.—Synchronizing the controls in the boiler-room was accomplished through an arrangement of valves, piping, and linkage so that the draught through each boiler was automatically adjusted to the quantity of steam generated and the amount of fuel consumed. The boilers had multiple services in the refinery, furnishing steam for the gasoline plant, and power for drilling wells, for oil-treating plants and clean-out operations in the field.

Detailed description of the arrangement is presented and illustrated.

Frequent tests of stock gases were made after the controls were installed in order to adjust the travel ranges of the controls. After the tests, it was found that the operation of the assembly became fully automatic and required no further adjustments, except where excessive wear of pins necessitated them. A. H. N.

160.* Caustic Embrittlement of Steam Boilers. W. S. Cazort. *Refiner*, May 1940, **19** (5), 156–158.—Caustic embrittlement is a series of intercrystalline breaks in seams and joints of boiler-plates, and particularly between rivet holes. This condition results from the conversion of sodium bicarbonate into sodium hydroxide, through the formation of the intermediary sodium carbonate, by the action of the heat supplied by the boiler. The caustic soda increases in concentration, and when it is in excess of 2 gm./litre under pressure and high-temperatures it causes this cracking between rivets and seams, with disastrous consequences.

Two agencies for the prevention of caustic embrittlement are soluble sulphate and phosphate salts. For every boiler pressure there is an optimum sulphate to carbonate ratio, ranging from 1 : 1 to 3 : 1 as pressure rises from 5 to 250 lb./sq. in., which will completely eliminate embrittlement by caustic soda. Most alkaline waters contain a certain amount of sulphates, thus only complementary quantities are required to raise the sulphate content to the required ratio.

The determination of alkalinity and the calculations involved in checking alkaline embrittlement are detailed and illustrated by an example which is worked out numerically. A. H. N.

161.* Gas Lift Combined with Gasoline Manufacture. Anon. *Refiner*, May 1940, **19** (5), 159–162.—A plant which was designed with a view to gas lift as well as gasoline manufacture also deals with a varying load of gas the peak load of which is 12,000,000 cu. ft./day. The quality of this gas, in so far as gasoline content is concerned, varies by the method of producing the oil. Field tests indicate that the content ranges from 1 to 4 gal./1,000,000 cu. ft., but the average is around 1·20 gal. Content of the gas may range from 1 gal. up to 4 gal., depending on the class of wells from which the gas is being taken, and also on the manner in which the wells are produced, whether they are opened wide for the daily allowable, or produced in a more orderly manner. On an average, the plant produces around 1·10 gal./1,000,000 cu. ft. of 18 lb. gasoline, and about 1·00 gal./1,000,000 cu. ft. of butane, the two varying according to whether the main product of gasoline requires a larger or smaller quantity of butanes for vapour-pressure control.

The gas is received through three trunk vacuum lines. The vacuum is held down as low as possible to bring in the gas without excessive back pressure on the field equipment, and so that a minimum amount of air will be drawn into the gathering system. A high vacuum is most detrimental in a field of this nature where quantities of H_2S are contained in the gas, due to the higher rates of corrosion of equipment when this sulphur compound is acted upon by oxygen and passed through iron or steel pipes and vessels. A. H. N.

162.* Selection of the Most Economical Pipe and Valve Size and Rate of Flow in Piping Systems. S. P. Johnson and F. L. Maker. *Refiner*, June 1940, **19** (6), 169–180. *Paper Presented before American Petroleum Institute.*—The phase of the subject covered by this paper is limited to the proper choice of pipe size to give a balanced and economic cost value. The size of long pipe-lines is usually determined by balancing pipe cost against pumping cost. As a formal economic study for each element of a plant piping system would be out of the question, various shortcut methods are commonly employed, the most usual being to assume a reasonable velocity or a reasonable pressure drop on the basis of experience or common sense. This paper examines these methods and shows that they are apt to err when the density or viscosity of the fluid is unusual or when special materials are required. A rational method of selecting pipe size for any fluid is worked out, and charts are included which permit the economic selection of size to be made with a minimum of calculation.

It is shown that valves, and sometimes other appurtenances, are not necessarily best made the same size as the line. The economy of small-size valves has been worked out, and suggestions are made for installation.

The flow rate in a piping system is usually dictated by process requirements or basis assumptions. There are, however, interesting exceptions when the designer of the piping must chose the flow rate as well as the pipe size to secure the lowest overall cost. One of the most important problems of this class arises in designing a loading system for tank-ships, tank-cars, or trucks when a three-way balance should be struck between pipe cost, pumping cost, and cost of delay. This problem is simplified by the development of a chart showing the most economical flow rate for any given or assumed loading system. A. H. N.

163.* Application of Superheated Steam to Combination-Unit Prime Movers. A. E. Harnsberger. *Refiner*, June 1940, **19** (6), 181–184. *Paper Presented before American Petroleum Institute.*—Combination cracking units of large capacity, involving as they do heavy pumping loads, present a power problem to the refiner. This paper summarizes an investigation made by a refiner on the best method to meet the power requirements of a 25,000-brl. crude-charging-capacity combination unit. It was necessary to adapt the installation to meet the limited steam-production facilities in an existing plant.

The investigation revealed the practicability of generating steam from waste heat. By superheating the steam to the largest power-consuming units, and by condensing the steam from these units under vacuum, it was possible to reduce the steam requirements and make a boiler-house extension unnecessary. Operating data are given showing that original design expectations are being achieved. A. H. N.

164.* Removal of Sulphuric-Acid Fog by Bubble-Phase Absorption. D. W. Bransky and F. F. Diwoky. *Refiner*, June 1940, **19** (6), 191–195. *Paper Presented before American Petroleum Institute.*—Various methods of the removal of H_2SO_4 droplets from the stack gases of sulphuric-acid manufacturing and concentrating plants have been evaluated. The various known methods have been divided into two categories, viz., effective and ineffective, according to the practicability of the method from both the viewpoint of sulphuric-acid removal and the economic picture. Effective methods such as (1) the filtration of the stack gases through various wetted media ; (2) the decomposition of acid mist by heating to 1000° F. or above under controlled conditions of combustion ; (3) the use of Cottrell precipitators ; (4) the use of steam ejectors with subsequent condensation of the steam ; and (5) the use of turbo-mixers were discarded because of high investment or high operating costs. A new method, in which the gas is bubbled through an aqueous solution of a foam-producing substance, has been found to be extremely effective in removing H_2SO_4 droplets, and has a very low investment cost.

The most effective foam-forming agent found consists of the sulphonic acids obtained from the hydrolysis product of the acid sludge derived from the treatment of petroleum. These acids are readily soluble in water, form a relatively stable bubble phase in a dilute aqueous or sulphuric-acid solution, and are soluble in aqueous solutions of H_2SO_4 containing up to 10% acidity. A laboratory investigation subsequently followed by a pilot-plant investigation indicated that the method was effective in removing approximately 90–98% of the H_2SO_4 in the incoming gas, and that the efficiency of the H_2SO_4 removal was practically independent of the concentration of the H_2SO_4 in the gas and the temperature of the solution over ranges of 80–186° F. It was found also that the efficiency of removal was affected greatly by contact time. The pilot-plant experiments demonstrated that, with a contact time of 10 sec. to 11 sec., approximately 93–95% of the total H_2SO_4 may be removed in continuous equipment having a pressure drop of approximately 2 in. of water. Economic calculations have indicated installations, operating, and repair cost to be approximately 50% that of equivalent electrical precipitation equipment. A. H. N.

165.* Chemical Removal of Scale From Refinery Equipment. L. W. Lee. *Refiner*, June 1940, **19** (6), 222–224.—To be assured of successful results by chemical removal of industrial scale deposits from heat exchangers, cooling coils, condensers, I.C.E. cooling jackets, and other types of equipments, it is necessary to know the exact composition of the scale. The usual tests are carried out with portable solubility test apparatus ; in more difficult cases *X*-ray powder diffraction patterns are obtained for the determination of chemical composition of the crystalline material. The same crystalline material gives the same pattern, which is unique to the crystal. From these tests the solvent to be used is determined.

The manner of introduction of the solvent is very important ; best results have been obtained by introducing fresh solvent into the vessel to be serviced by as many avenues as possible. The solvent is circulated back to the tank and again into the equipment.

A recent type of heat-exchanger departs from the conventional type by having spines extending from the pipe in great numbers, so as to increase the heat-exchanging area by as much as 20 times the normal. The removal of scale and sludge from such an irregular and spiky surface is particularly adaptable to chemical treatment in preference to mechanical.

Tower trays and other similar equipment can be descaled in some 36 hr.—a much shorter time than would be possible if the equipment were dismantled and cleaned mechanically. A. H. N.

166.* Rolling-in of Upset and Close Tolerance Machined Tools for Cracking Furnace Installations. F. C. Braun and M. Fleischmann. *Refiner*, July 1940, **19** (7), 225–231.—In order to gain information in regard to certain variables in the rolling-in technique which may affect tightness and safety of rolled joints, three big companies conducted an investigation, the conclusions of which were :—

The header material should be considerably harder than the tubing, to assure well-developed shear points. It is suggested that headers for the installation of alloy tubes with a maximum hardness of 163 Brinell should be heat-treated to a hardness of 220 Brinell minimum. In general, it is recommended that the hardness of the header should exceed the hardness of the tubes by a minimum of 60 Brinell numbers.

There is an optimum amount of expansion for each tube size which will produce best shear points and tightest fit. Not only insufficient rolling, but also too much rolling will cause joints of doubtful quality. Too much expansion of the tube into the tube hole will result in an excessive flow of the tube material out of the header, with the danger of shearing off proper shear points which already have been formed.

The use of wide-angle flaring rolls on the expander tool should be discouraged, as the flaring of the tube ends will cause a longitudinal flow of the tube material, injuring satisfactory shear points formed by the expander rolls.

The procedure and test results of the investigations are reported, and a chart indicates recommended expansion for a given size tube end, upset and machined to close tolerance. A new rolling-in tool is described and illustrated. A. H. N.

167.* Refrigeration and Dehydration Fit Plant into Repressuring System. Anon. *Refiner*, July 1940, **19** (7), 235–239.—In order to fit a natural gasoline-manufacturing plant into the repressuring programme of a field, compression, refrigeration, and dehydration have been incorporated in the plant.

Three stages of compression are used. In the first, gas is drawn from the field and boosted to around 40 lb./sq. in. gauge. In the next it is increased to 300 lb., while the third is to 1800 lb. for formation repressuring.

The first stage is typical gasoline-plant practice, drawing the gas through the gathering system at the field pressure, and boosting it to 40 lb. gauge. The gas is cooled in atmospheric sections set in a water-cooling tower and scrubbed to remove liquids produced. The condensate accumulated in this stage consists of high-boiling fractions which are combined with final production for further processing. No attempt is made to reduce the temperature of the gas at this low pressure below that obtained in conventional atmospheric-section cooling, and the gas is then picked up by the intermediate cylinders, which raise the pressure to 300 lb. gauge. The intermediate cooling is accomplished in atmospheric cooling sections, and the gas and condensate then enter the refrigeration and glycol dehydration units without separation.

The refrigeration side of this plant is handled by expanded propane contained in a closed system and recycled, much the same as other plants use *iso*butane or a rough cut of propane-butane from the stabilizer reflux when cooling absorption oil and commodities when atmospheric conditions are unfavourable for low temperatures by ordinary water cooling. Two horizontal gas engines compress the gas and deliver it to atmospheric condensers with the liquid product passing to a surge-tank. From this tank the liquid passes to the propane flash-tank, and from there to the propane chiller, counter-current to the stream of gas, gasoline, and glycol, and thus, when returned to the engines, completes the cycle. A. H. N.

168.* Aviation Gasoline Plant Near the Arctic Circle. R. E. Parkhurst. *Refiner*, July 1940, **19** (7), 240–245.—The most northerly refinery in the world has recently been completed within 100 miles of the Arctic Circle. Many difficulties were encountered and some of these are recounted.

The means of transportation imposed certain physical restrictions due to the size of the barges and type of trucks operating on the Fort Fitzgerald–Fort Smith portage road—no single piece could be over 10 tons in weight or occupy more space than 10 × 10 × 35 ft. long. Some welding was done on pipe supports, but generally all equipment had to be bolted, contrary to general practice. Maximum permissible pipe diameter was 4 in., on account of absence of facilities at the site for handling larger sizes. Some local lumber was available, but the general use of wood was ruled out. Even gin-poles for erection were shipped in. Because of scarcity of local skilled labour and a round trip transportation cost of $420 per man, and in order to reduce erection time to the minimum, it was necessary to assemble all equipment in the shop, so far as possible. Since Arctic weather conditions and open water would permit operation of the refinery only 90 days a year, the refiner desired as low an initial cost as possible for the installation, combined with low maintenance costs. Many other difficulties were due to the fact that the frost-line is some 50 ft. below ground and the ground never thaws out.

The plant was designed for three alternate operations, producing a stabilized gasoline of 270° F. Engler F.B.P., sidecut of light diesel fuel oil, and heavy fuel-oil bottoms for stationary diesels with approximate yields, respectively, of 19%, 41%, and 29%. The sidecut can be eliminated, running all the fuel oil as bottoms. It is also possible to run a 400° F. end-point motor gasoline overhead and take diesel fuel oil as bottoms.

A. H. N.

169.* New Principle in Boiler Feed-Water Level Control. J. A. Campbell. *Refiner*, July 1940, **19** (7), 246–249.—It was reported in a previous paper (*Refiner*, February 1937, p. 60) that good results were obtained from a thermostatic-tube type of boiler-feed regulator. In the previous report an inclined brass tube was set up, 48 in. long and ¾ in. in diameter, and alongside of it a gauge glass with thermometer well at each end. The upper end of the tube was connected into the steam space at the top of the water column, and the lower end to the water column near the bottom, well below the water level. These connections permitted the water to seek its own level in the tube. This level checked lower than the level in the glass column, which is always lower than the actual level in the boiler. Two reasons are given for this : (1) frothing of water in the boiler by the steam bubble reduces the density of the water there by some 15% ; (2) the temperature effect.

In this paper a development of a still better and very sensitive regulator is given. An interesting part is where the expansion is multiplied by using a geometrical truth. A tube which gets hot and cold, and thus regulates a valve travel, is incorporated in a triangular set of bars. It is a principle in geometry that a triangle, so long as its legs remain straight, cannot be changed in shape without changing the length of one of the legs. When the tube gets hotter, due to falling water level, it lengthens. It cannot lengthen along its own axis because of the restraining bar, which remains of fixed length, so the outer end of the tube must swing upward on the arc. This upward movement, due to the ratio of tube length to the spread between the two anchored ends, is six times as great as the actual increase in tube length due to expansion. The arc having a radius of 42 in. and the movement of the outer end casing along the arc being relatively small, for all practical purposes, must be regarded as a vertical movement. From cold tube to steam-filled tube at 150 lb. pressure this vertical movement is 1 in.

A. H. N.

170.* Graphical Methods Applicable to Refinery Problems. J. W. Greene. *Refiner*, August 1940, **19** (8), 268–274.—The paper is an adaptation of simple graphical interpretation of elementary differential and integral calculus to problems met in the refinery, and is based on the fundamental concepts that the slope of a curve is its differential coefficient and the area bounded by a curve and two ordinates at two abscissa values and the x-axis is its integral between the limits of the abscissa. An example on enthalpy and heat capacity illustrates the use of graphical differentiation and integration.

Three methods are used to illustrate an integration example in the design of an absorption tower where the volume of the tower is required.

Solution of quadratic and cubic equations graphically by reducing the problem to two functions which, when plotted, yield a curve and a straight line intersecting at the roots of the equation in the usual manner forms part of the paper.

It is noted that if average conditions are used, instead of point conditions accurately determined, errors will be introduced into the calculations.

The paper is clearly written and well illustrated. A. H. N.

171.* Annealing Furnace Permits Re-Use of Still Tubes. Anon. *Refiner*, August 1940, **19** (8), 280–281.—An annealing furnace is described which was designed for reconditioning equipment in a refinery, after welding ends on to tubes to replace parts damaged by corrosion and erosion. After welding, the tubes are swung by cranes on to an oven, which is controlled by three thermocouples suitably placed, and all openings in the super-structure are closed by plastic insulation similar to that applied to a boiler by mixing with water and trowelling on. The temperature is gradually raised throughout the oven, and at least two-thirds of the lengths of the tubes are at a uniform temperature.

Photographs illustrate various aspects of the process and the furnace. A. H. N.

172.* Fractionation Concentrated at One Plant. Anon. *Refiner*, August 1940, **19** (8), 297–298.—A plant is described where all fractionating equipment are grouped at one point, the gasolines manufactured in various plants being pumped to this centralized fractionation unit. Feed control and arrangement as well as reconditioning systems and blending operations are described. A. H. N.

173.* Suspended Walls. H. O. Johanson. *Refiner*, August 1940, **19** (8), 299–300.—Five reasons are given for the failure of a refractory lined wall: melting, softening, spalling, fluxing, and expansion. Suspended walls greatly reduce, and in many cases entirely eliminate, the causes of these failures, and thus most refinery oil-stills and boilers are installed with suspended walls.

There are only rare cases of failure by melting, and many failures which appear to be due to melting are in reality due to fluxing.

Softening is, on the other hand, a major cause of trouble. The bricks near the bottom of the furnace wall soften, and the result may be the collapse of the inner surface of the furnace. Air-cooled suspended walls improve this condition, as the lower bricks are not raised to the softening temperature and do not carry heavy weights. The walls should be thin, and for air-cooling should not exceed 10 in.

Spalling is the effect produced by alternately heating and cooling the fire-bricks. Suspended walls do not suffer badly, as the bricks are not constrained.

Fluxing results from the interaction of the ash with the firebrick, with the result of forming a fluid, at the high temperatures operating, and washing away the bricks. This appears as if failure were by melting. Air-cooled suspended walls again remove this trouble, as solid ash cannot react with the bricks. Excess air in the fuel is another remedy, but the stack losses are then high.

Expansion troubles in ordinary walls are due to the fact that the wall behaves monolithically. In suspended walls each section of the wall is independent of all other sections, and is therefore free to expand.

Sectional supported walls are briefly discussed, and usages in designs for boiler work and oil stills are outlined. Experimentation is still being carried out on various designs. A. H. N.

174.* Processes in Refining Petroleum. Anon. *Refiner*, September 1940, **19** (9), 313–353.—A section on process development is presented in which a flowsheet and a page of description are given for each process which is either newly developed in 1940, or which has shown material changes either in flow diagram or description during the same period. The information is compiled either from the holder of the patent or from the concern privileged to license the process.

The following processes are described and illustrated: Badger Distillation Processes;

Bari-Sol Process (centrifugal dewaxing); Duo-Sol Solvent Extraction Process; Filtrol Fractionation Process (highly activated bleaching clay used); Speciality Absorption Process (for propane); Furfural Refining Process (solvent refining of lubricating oils); High-Pressure Gas Recycling Process (for pressure-maintenance in distillate fields); Houdry Catalytic Cracking Process (high octane gasoline and aviation fuel); Houdry Catalytic Treating Process (vapour phase); Polyform or Gas Reversion (see Abstract No. 140); Perco Catalytic Desulphurization; Perco Copper Sweetening Processes; Pure Oil Thermal Polymerization (high-octane blending stock from gases); Sharples Dewaxing Process (centrifugal dewaxing); Shell Phosphate Process (removal and recovery of H_2S); Regenerative Mercaptan Removal; Shell Solutizer Process (sweetening gasoline); Sulphuric Acid Alkylation Process; Catalytic Polymerization Process (U.O.P. Midget and large scale plants are described); and X-YTE Percolation Filtration Process. A. H. N.

175.* Depropanization of Butane. L. J. Coulthurst. *Oil Gas J.*, 11.7.40, **39** (9), 50; *Nat. Pet. News*, 10.7.40, **32** (28), 50.—Propane is frequently present in gasoline in insufficient quantities to justify the installation of a depropanizer, and is lost during handling and storage, taking with it some of the more valuable hydrocarbons. The extent to which large percentages of butane can substitute small percentages of propane to give gasolines of required vapour pressure is discussed. For final vapour pressures of 8, 10, 12, and 14 lb. respectively, 1 brl. of propane may be substituted by 4·7, 4·9, 5·1, and 5·3 brl., respectively, of butane. In such cases the butane possesses gasoline value. In one case the installation of a catalytic polymerization unit, removing the propylene and butylenes, gave a sufficiently high propane concentration in the residual butane to warrant the installation of a butane depropanizer. The substitution of propane by this propane-free butane increased the gasoline production sufficiently to make the installation an economic success. C. L. G.

176.* Doctor Test has Indirect Value. W. A. Schulze. *Nat. Petrol. News*, 7.8.40, **32** (32), R. 290.—The article suggests that although the consumer is less conscious of odour as a significant property of gasoline, sweetening processes are still worth while. Data are presented to show that the gum stability of a sweetened spirit is greater than that of a sour gasoline, provided that the sweetening process does not remove natural inhibitors. Sour gasolines are shown to suffer a loss in octane number when exposed to sunlight, a factor stated to be of significance when the gasoline is dispensed from " visible bowl " pumps. The " Doctor Test " is defended as safety device for the refiner. H. G.

177.* New Dehydration-Refrigeration Plants Involve Use of Diethylene Glycol. H. L. Flood. *Petrol. Engr*, May 1940, **11** (8), 113.—The use of diethylene glycol as a dehydrating agent in gasoline plants operated in connection with repressuring operation so that the efficiency of the glycol is increased by operating at a lower temperature whilst the gasoline yield is also increased.

The gas is first scrubbed and compressed in two stages to 300 lb., separated water and condensed gasoline being drawn off. Diethylene glycol is then injected into the gas stream, which is subjected to initial chilling in a series of heat exchangers and then passed into a propane chiller. The gas is then passed to a main gasoline separator at 300 lb. and then through three further separators at 135 lb., 70 lb., and 50 lb., respectively. Absorption of water by the diethylene glycol occurs continuously during the chilling and separation processes. The dilute glycol–gasoline mixture is settled in the last separator, the former being pumped into raw gasoline storage and subsequently stabilized, and the latter drawn off into a glycol still, where it is reconcentrated. The gas is compressed to 1800 lb. and returned to the field. It has a dew-point of 40° F., and this is considered to be satisfactory for hydrate-free operation during the greater part of the year. R. J. E.

178.* Developments in the removal of H_2S From Natural and Refinery Gases. W. A. Leech. *Petrol. Engr*, Mid-Year 1940, **11** (10), 97.—A wide variety of processes for the removal of H_2S from gas is now available, some providing by-products of sufficient value to pay for the cost of purification. Recently installed plants utilize the following processes: (1) absorption in amines, in one case incorporating diethylene glycol dehydration; (2) absorption in potassium phosphate, removing 95% of the H_2S from a sour gas; and (3) treatment with iron oxide (this being considered the simplest and cheapest means of purifying small quantities of low H_2S content gas in isolated locations). Several refineries have installed processes for the recovery of H_2S as sulphur or sulphuric acid (for use in alkylation processes, etc.). C. L. G.

179.* Effect of Atmospheric Conditions on Diesel Engine Performance. J. S. Doolittle. *Oil Gas J.*, 1.8.40, **39** (12), 42.—A study has been made of the effect of variations in pressure, temperature, and humidity of the air on the performance of a two-cycle diesel engine, running under at least 10 different loads varying from 30% rating to the maximum possible with steady operation. Humidity effects were examined with intake air at 80° F. and at 48% and 90% humidity, and at 120° F. with 27% and 90% humidity, and temperature effects with air at 45° F., 80° F., and 120° F. Intake pressures were varied from 22·5 to 33·5 in. Hg at constant exhaust pressure, and exhaust pressures from 29 to 31 in. Hg at constant intake pressures. In other tests equal intake and exhaust pressures from 29·4 to 31 in. Hg were used. The tests indicated that at loads lower than the load of minimum fuel rate, changes in air density as high as 15%, resulting from variations in any or all of the factors examined, have little effect on the fuel rate. With loads in excess of this, changes in intake air pressure, temperature, humidty, or exhaust pressure affect the amount of air taken into the cylinder, and hence influence both the fuel rate and the maximum power output. Further, if the pressure differential between the scavenging air and main cylinder pressure is abnormally small, changes in either the exhaust or intake pressures will affect the air supply to the engine, and hence fuel rate and maximum power output. C. L. G.

180.* Conserving Lubricating Oil. C. I. Kelly. *Petroleum*, August 1940, **2** (4), 111–122.—This is a plea for the recovery of used lubricating oils on a national basis.

The author is of the opinion that while the nation is fully conscious of the part played by liquid fuels in the national life, and of the necessity of conserving supplies, it does not realize the important part played by lubricating oil. This may in part be due to the fact that whilst liquid fuels amount to 80% or more of the total petroleum imports, lubricating oil represents only 3·5% of these imports.

Used oils can be reclaimed and refined into oils indistinguishable from new straight mineral oils at a cost lower than the new oil in war-time. Two processes are discussed: (1) that using activated earth followed by filtration and sometimes by removal of diluent; (2) a chemical treatment with H_2SO_4 followed by neutralization and earth treatment.

Two types of small recovery plant are described, both using the activated-earth treatment, and analyses of aero oils recovered by these methods are compared with the requirements of the DTD. 109 specification.

The question of the collection of crankcase drainings for recovery treatment is discussed. D. L. S.

181.* Recovery Progress. A. T. Wilford. *Petroleum*, August 1940, **2** (4), 123.—The heavier types of lubricating oils, particularly if badly contaminated, are not suitable for recovery. These, however, constitute only a fraction of the total volume of waste lubricants, which are mainly derived from I.C. engine crankcases and which are of medium or light viscosity.

While at first sight it might seem logical to return used oil to the refineries for treatment, the problem of the transportation of relatively small quantities of different grades would arise. There is much to be said in favour of the installation of small plants at individual garages or groups of garages.

The recovery treatment necessary depends to some extent on the condition of the oil, and thus the frequency with which the oil is changed. Simple filtration can be used quite successfully in some cases. In others, however, it is necessary to top, earth treat with or without a preliminary acid treatment, and then filter. Steps are being taken by London Transport to reclaim oil both from petrol engines and C.I. engines, and the results obtained so far are regarded as satisfactory. D. L. S.

182. Application of Welding to the Petroleum Industry. M. Couturier. *Sci. et Ind.*, 1940, 290 bis (Supplement to *Revue Mechanique*), 92–98.—Welding has found application in the construction of fractionating towers, storage tanks, liquid gas holders, pipe-lines, etc. The applications of the process to a variety of petroleum engineering problems are reviewed. T. C. G. T.

183. Struggle Against Corrosion in the Petroleum Industry. G. Daric. *Sci. et Ind.*, 1940, 290 bis (Supplement to *Revue Mechanique*), 86–91.—This paper gives an account of the various forms of corrosion—chemical, electrolytic, galvanic, etc.—and the symptoms observed from each, together with the mechanism of their action and the precautions to be taken. It also reviews corrosion occurring in underground pipelines, storage tanks, and corrosion in cracking tubes operating under conditions of high temperatures and pressures and, in conclusion, considers corrosion-resisting steels. R. D. S.

184.* Acid Clay and Activated Clay in Japan. Anon. *J. Soc. Chem. Ind.* (*Japan*), 1940, **43** (10), 303 B.—The principal deposits of acid clay are found in the north of Japan, and are mined in the usual way—viz. the raw acid clay is dug out with pick and shovel in the open air and conveyed on trucks to a nearby factory, where it is crushed into lumps and dried by direct heat in iron pans. The geological origin of Japanese acid clay is obscure, but it is thought to be produced by the natural decomposition of a certain felspar which is contained in igneous rocks such as liparite. Owing to the absorptive and catalytic actions of the clay, it has many industrial applications, and is employed in the following processes : the recovery of natural gasoline from natural gas ; decolorization of mineral and fatty oils ; removal of sulphur compounds from industrial gases, mineral oils and solvents ; dehydration of alcohol ; preparation of cracked gasoline from heavy oils and the preparation of lubricating oils and artificial resins from cracked oils derived from mineral and fatty oils. A highly efficient activated clay has been made from the natural acid clay which is extensively used in the refining of high-grade lubricating oils. B. M. H. T.

185. Patents on Refining and Refinery Plant. N.V. De Bataafsche Petroleum Maatschippij. E.P. 528,610, 1.11.40. Appl. 18.5.38. Method of refining hydrocarbon mixtures in the vapour phase with sulphuric acid and/or water-soluble hydrogen sulphates. The hydrocarbon vapours are contacted at an elevated temperature with dilute aqueous solutions of sulphuric acid and/or hydrosulphates in quantities which are practically completely converted into sludge during the reaction.

N.V. De Bataafsche Petroleum Maatschippij. E.P. 528,611, 1.11.40. Appl. 10.5.39. Process for refining hydrocarbon mixtures in the vapour phase with organic sulphonic acid. The hydrocarbon vapours are contacted at an elevated temperature with a dilute solution of an organic sulphonic acid, if necessary together with sulphuric acid, in a chemically inert solvent which is volatile under the reaction conditions.

Standard Oil Development Corp. E.P. 528,766, 6.11.40. Appl. 5.5.39. Method of revivifying spent decolorizing adsorbent used in the decolorization of oils. The adsorbent is treated with an aqueous treating liquid, which is immiscible with the liquid adsorbed on the adsorbent, at a temperature between 300° and 710° F., and under a pressure sufficient to maintain the treating liquid in the liquid phase throughout the operation.

Standard Oil Development Corp. E.P. 528,919, 11.11.40. Appl. 8.5.39. Improvements in design of apparatus for carrying out reactions between finely divided solids and fluids. The improvements relate more particularly to apparatus for the cracking of petroleum hydrocarbons with the aid of a finely divided solid catalyst and to the subsequent regeneration of the catalyst.

N.V. De Bataafsche Petroleum Maatschippij. E.P. 529,210, 15.11.40. Appl. 24.5.39. Process for splitting up two or more chemically related organic substances into two components or groups of components by treatment with two liquids, led in counter-current, which are mutually immiscible or miscible only to a slight extent. Examples of mixtures which can be split up are mixtures of nitrogen bases which can be separated with sulphuric acid from the acid sludge formed in the treatment of gasolines obtained by cracking special mineral oils.

Manchester Oxide Co., Ltd. E.P. 529,455, 21.11.40. Appl. 26.5.39. Process for treating spent sulphuric acid from petroleum refining by the use of a solvent consisting of or containing a substantial proportion of an aromatic hydrocarbon. The spent acid is treated simultaneously with the solvent and with water.

N.V. De Bataafsche Petroleum Maatschappij. E.P. 529,873, 29.11.40. Appl. 9.6.39. Process for refining hydrocarbons, preferably in the vapour phase, with phosphoric acid. Cracked benzene, fuel oil, and other distillates treated in accordance with the invention obtain a good colour, high oxidation stability, and show little or no tendency to gum formation.

N.V. de Bataafsche Petroleum Maatschappij. E.P. 529,997, 3.12.40. Appl. 31.5.39. Removal of mercaptans from hydrocarbon oils by treatment with an aqueous solution of caustic alkali. The invention has particular reference to the use of aqueous solutions of alkali metal hydroxides containing substantial amounts of alkali metal alkyl phenolates for carrying out the treatment.

H. G. Vesterdal. U.S.P. 2,216,968, 8.10.40. Appl. 28.5.40. Refining heavy hydrocarbon oils by treating with dilute aqueous caustic soda under sufficient pressure to maintain the oil and caustic-soda-solution liquid at the operating temperature. Thereafter the dilute caustic-soda solution is removed, together with the extracted material from the oil by decantation.

A. Shapiro. U.S.P. 2,217,387, 8.10.40. Appl. 24.7.39. During demulsification of petroleum the hydrogen-ion concentration of the water is brought within the range p_H 5·5–p_H 4·8 by adding to the emulsion a reagent capable of neutralizing excess acidity or alkalinity.

C. E. Dolbear. U.S.P. 2,221,183, 12.11.40. Appl. 24.4.36. Method of sweetening petroleum distillate by contacting with a reactive material selected from the group consisting of mercuric and cadmium oxides. A chemical conversion of "sour" mercaptan is thus caused. The distillate is then removed from contact with the reactive material and contacted with a divided solid adsorptive base-exchange zeolite to cause removal of dissolved mercaptides. Finally the distillate is separated from the zeolite and removed mercaptides.

E. Scheller. U.S.P. 2,221,559, 12.11.40. Appl. 20.10.37. Bleaching of oils, fats, and waxes by contacting in the emulsified state with an oxidizing agent in order to effect a partial bleach. Thereafter an acid is added in an amount sufficient to break the emulsion and an alkaline agent added to the product in sufficient amount to neutralize the free organic acid present. Finally the product is bleached in the liquid or molten state with hydrogen peroxide.

B. Malishev. U.S.P. 2,223,524, 3.12.40. Appl. 12.8.39. Refining cracked gasoline by treating with a catalyst consisting of a solid calcined product of a mixture of a substance taken from the group consisting of a mineral phosphate, superphosphate, and Thomas slag, with sulphuric acid. The treatment is conducted at a temperature between 150° and 300° C. and at a pressure sufficient to maintain the liquid state.

H. B. M.

Chemistry and Physics of Petroleum.

186.* Elements of Vaporization. The Phase Rule. Part I. R. L. Huntington. *Refiner*, January 1940, **19** (1), 16–18.—The phase rule is defined and the general equation $F + P = C + 2$ is explained. To illustrate the application of the phase rule, examples are worked out in full for a steam boiler and a mixture of gases.

It is explained that this series of papers has been prepared for a three-fold purpose: (1) to provide the man in the plant or refinery, who has had a year or two of college work, with certain tools and methods needed in the solution of engineering problems; (2) to enable the graduate engineer to review some of the principles he learnt in college; (3) to give the plant man who is planning on returning to college or studying in vocational classes a chance to acquire some skill and practice in making quantitative calculations. A. H. N.

187.* Can the Hydroxylation Theory of Combustion be Applied to Modern Furnaces? J. R. Darnell. *Refiner*, February 1940, **19** (2), 53–54.—The paper suggests that the answer to the title is "no," and further suggests that the question is worthy of research. The theory of hydroxylation assumes that there is a progressive oxidation of the hydrocarbons by intermediate steps, the first of which is the formation of the hydroxyl radical OH in combination with the carbon and hydrogen atoms to produce first an alcohol, then an aldehyde, and next an acid. The end-products, of course, are carbon dioxide and water vapour.

The chief criticism of this theory appears to be the fact that the experiments on which the theory is based were conducted at temperatures far below those operating in a modern commercial furnace. A. H. N.

188.* Partial Oxidation of Petroleum and Natural Gas. W. L. Faith. *Refiner*, March 1940, **19** (3), 68–72.—The present status of the partial oxidation of petroleum hydrocarbons is reviewed. The effect of the variables time, temperature, pressure, concentration, and catalysis on the reaction is emphasized. Problems involved in equipment design are also presented.

Of the processes being operated commercially, few operating details are available. One major oil company is manufacturing carload lots of methanol and formaldehyde by the oxidation of natural gas. Operating conditions probably include pressure in the neighbourhood of 250–350 lb./sq. in., elevated temperature, and a low oxygen–hydrocarbon ratio. Several metals and oxides, including platinum, are known to catalyze this reaction, but it is possible that the chemical nature of the surface is not nearly so important as time of contact.

Another commercial process is the oxidation of ethylene to ethylene oxide. Patent literature indicates that a catalyst consisting primarily of silver is used, that pressures up to 50 atmospheres, and temperatures ranging from 300° to 400° C. are employed.

In Europe, fatty acids capable of being converted into soaps are made by the oxidation of the heavier hydrocarbon fractions. Low-temperature liquid-phase processes are used both with and without catalysts. The products are converted into an inferior grade of soap. A. H. N.

189.* Elements of Vaporization and Condensation. Part II. R. L. Huntington. *Refiner*, March 1940, **19** (3), 80–84.—The application of phase-equilibria principles to the behaviour of a water–pentane mixture and mixture of two or more hydrocarbons under vaporization conditions is studied by means of hypothetical, constant-pressure, frictionless piston overlying such systems in closed cylinders.

Raoult's and Henry's Laws are studied and illustrated. The case of the exception of water and propane mixtures is cited.

Water is slightly soluble in practically all hydrocarbons. In the fractionation of propane a small amount of dissolved water is often found to be present. The removal of this water from the propane is required before it can be sold as domestic fuel, as the presence of water is apt to cause freezing in the gas-reducing regulators. Drying agents such as silica gel have been used for the dehydration of propane, but the most satisfactory method of water removal has been effected through fractionation. Although propane has almost 200 times the vapour pressure of water, dissolved water in propane does not behave according to Raoult's Law, but, on the contrary, the dissolved water proves to be more volatile than the propane. As a result the water is taken out overhead from the fractionating column. A. H. N.

190. *Elements of Vaporization and Condensation. Part III. R. L. Huntington. *Refiner*, April 1940, **19** (4), 125–126.—The calculation of evaporization lossos from large atmospheric storage tanks is discussed. Vapour temperatures in a tank will often go as high as 30° or 40° F. above the air temperature on clear, sunny days when there is little wind. At night, however, the air and vapour temperatures will tend to approach each other, so that the vapour may be only 2–10° F. above the air temperature, early in the morning just before sunrise. The high vapour temperature at noon day, or shortly thereafter, is the result of the conversion of radiant energy into heat energy on the surface of the tank, which may reach a temperature of 140–150° F.—some 20–30° F. higher than the vapour inside the tank. The loss of heat, by radiation and convection, from the tank to the cooler surroundings cannot begin to keep up with the rapid absorption of radiant energy from the sun when it is high in the sky.

The liquid in the tank picks up some radiant energy from the sun through the walls of the tank; however, the far greater mass and heat capacity of the liquid causes this phase to lag behind in temperature change. Consequently, the liquid in a tank may vary only 2° or 3° F. between day and night. Vaporization in the day-time tends to cool the liquid, and condensation of part of the gasoline vapour has a heating effect at night, both these factors tending to keep the liquid constant in temperature over the 24 hours.

Losses are computed for a typical example.

It has been found from experience, however, that breathing losses increase up to liquid withdrawal of approximately 10%, above which the losses are practically constant. This phenomenon is due to the fact that the air does not mix very readily with the vapour in tanks containing a small amount of liquid, since the vapour is usually 2–4 times as dense as the atmosphere. A. H. N.

191.* Deviation of Natural Gas from Boyle's Law. F. H. Dotterweich. *Refiner*, May 1940, **19** (5), 148–150.—The correction factor in compressing natural gas is given by the formula, $N = \frac{0{\cdot}154\,p(m + 4l + 3c + 0{\cdot}22a)}{1000}$ where N = deviation in per cent., p = pressure in lb./sq. in. gauge, m = per cent. methane, e = per cent. ethane, c = per cent. carbon dioxide, and a = per cent. air. It is suggested that gas volume obtained from flow measurements at high pressures be multiplied by a correction factor of the square root of $(1 + N/100)$; if the measurement is by a displacement meter, the correction factor is $(1 + N/100)$.

Deviation factors giving the deviation of the behaviour of natural gas from Boyle's Law are plotted on pressure-deviation factor graphs as isotherms at temperatures between 32° F. and 212° F. The graphs show maxima at nearly 160 atmospheres, coincide at nearly 480 atmospheres for all temperatures, and continue to yield decreasing values beyond.

Correction factors are also discussed at reduced pressures and temperatures and general graphs for PV/RT at various values of P_R show better agreement with the gas laws at values of T_R increase from 1, the critical temperature, to 1·70.

Methods of calculations and their range of applicability and limitations are illustrated by a numerical example. A. H. N.

192.* Elements of Vaporization and Condensation. Part IV. R. L. Huntington. *Refiner*, May 1940, **19** (5), 164–166.—Flash or equilibrium distribution, batch or differential vaporization, and combination flash and differential vaporization are discussed. An empirical relationship, such as the one which has been worked out for the flashing of crude oils, is needed for the simplification of calculations for stage separation, but to date no satisfactory correlation of this nature has been developed. In previous papers of this series the method of calculating the separation of two component mixtures of hydrocarbons has been explained. Although the same principles are involved in the handling of three or more hydrocarbons, the calculation requires far more labour, as the trial-and-error or successive-approximation method is the only feasible attack to make. A straightforward algebraic solution of a three-component mixture is possible, but it calls for a far greater mass of calculations than the trial-and-error method.

A method of conducting material balance is explained, and an example, worked out completely by trial and error, illustrates calculation of bubble point of a gas mixture of propane, and normal butane and pentane. A. H. N.

193.* Elements of Vaporization and Condensation. Part V. R. L. Huntington. *Refiner*, June 1940, **19** (6), 219–221.—The utility of phase diagrams in distillation studies is illustrated, and water, being a most commonly known fluid, is made the subject of detailed study as an illustration.

Graphs showing the isotherms, isochores, and isobars are given for water. However, the abscissa for the isotherms is marked "temperature" instead of "volume." The significance of the three diagrams and that of the triple point is briefly reviewed, and the three diagrams are combined in a solid P–V–T model. The application of the P–V diagram in power generation problems is indicated.

An interesting illustration of the principles involved is made by an example of gas-lifting water, under isothermal conditions, through a height of 55 ft., by a gas of a simple mixture of methane, ethane, and propane, and requirements of gas are calculated on the assumption of isothermal expansion and an efficiency of 40%. A. H. N.

194.* Mixed Catalysts. V. N. Ipatieff. *Refiner*, July 1940, **19** (7), 250–255.—Prof. Ipatieff reviews in this paper certain of his work and theories on the mechanism of catalysis, the rôle of promoters, and the action of poisons.

The necessity of hydrogen chloride in the alkylation of, say, benzene in the presence of aluminium or its chloride is attributed to the necessity of the formation of hydrogen aluminium chloride, which is the true catalyst of the reaction.

The well-known rôle of water, as postulated by Ipatieff and his co-workers, in the action of alumina on alcohols is reviewed.

The action of a mixed catalyst is similar to that of a promoter, in that it either improves the physical condition of the catalyst or evokes a new reaction which will help the other. Only small quantities are sometimes necessary to affect the rate of reaction. It is proposed to call the composition of maximum activity of a mixed catalyst the *eucoactive* composition.

The mechanism of poisoning is believed to be based on the fact that molecules of different chemical constitution than that of the catalyst will always be present in the catalyst—*i.e.*, a reduced metal catalyst will always contain the oxide, and *vice versa*. The introduction of a catalyst poisoner in negligible quantities can stop the action of a catalyst by weakening or paralyzing the action of the molecules producing a given process, in consequence of which fact the neighbouring molecules will not receive a sufficient amount of energy and the process will cease. A similarity from the animal kingdom is cited—a minute amount of poison will kill a large animal if a vital physiological process in a very limited place of the organism is stopped. Energy cannot be carried to other organs of the body.

An example is given from the inhibiting effect of a special mucus secreted by the gastric glands on the mixed catalyst existing in the stomachs of animals—pepsin and dilute hydrochloric acid—to illustrate the uniformity of laws governing catalytic reaction in the animal and vegetable as well as the mineral kingdom. A. H. N.

195.* Elements of Vaporization and Condensation. Part VI. R. L. Huntington. *Refiner*, July 1940, **19** (7), 256–259.—Three types of binary mixtures are considered in the study of their pressure–temperature–composition relationship: (1) mixtures in which two components are miscible in all proportions; (2) mixtures in which the two components are totally immiscible; and (3) mixtures which are miscible within certain ranges of composition but outside these ranges form two phases, each phase being a definite mixture of the two components.

The pressure–composition of each of the three types is illustrated graphically in the conventional manner, each case being studied by citing a typical example.

Maximum and minimum boiling-point mixtures are further studied and illustrated. A. H. N.

196.* Kinetics and Mechanism of Decomposition of Hydrocarbons. M. P. Eshevskaya. *Refiner*, August 1940, **19** (8), 264–267.—In a study of the kinetics of decomposition of lower paraffins under low pressures, it was shown in some individual cases that the reactions are retarded by the reaction products. In this study the inhibiting effect of certain unsaturated hydrocarbons on the decomposition of paraffin hydrocarbons, *iso*-octane being specifically studied, is reported for ethylene, propene, normal butene, *iso*butene, 2 : 2 : 4-trimethylpentene, bivinyl, *cyclo*hexene, and *iso*prene.

Di*iso*butene, normal butene, and ethylene exert no noticeable inhibition under atmospheric pressure on the decomposition of *iso*-octane.

*iso*Butene, propene, bivinyl, *cyclo*hexane and *iso*prene, on the other hand, greatly reduce the rate of reaction.

An increase in the concentration of propene or bivinyl, used as retarding agents, is established to result in raising the retarding effect at first rapidly and then more slowly.

The mechanism of the retarding action is explained on the basis that the average length of the chain in the decomposition of ethane is 100, and that certain of the free radicles combine with the retarding agent, thus reducing the length of the chain.

A. H. N.

197.* Elements of Vaporization and Condensation. Part VII. Distillation and Fractionation. R. L. Huntington. *Refiner*, August 1940, **19** (8), 287–290.—The fundamental principles involved in simple distillation and fractionation are outlined briefly. The advantages of the pipe-still over the shell-still are listed.

Bubble-plate fractionating columns are described in a general way, and the main points to bear in mind in designing the size, disposition, shape, and number of bubble caps, downspouts, weirs for overflow, etc., are given. The Souder–Mott–Brown formula for calculating permissible mass velocity of vapours in towers to avoid entrainment is given; *i.e.*:—

$$W = C[d_2(d_1 - d_2)]^{\frac{1}{2}}.$$

However, no values are given for the constant C to be used in the formula. References are included.

Two systems for fractionating a mixture of propane, normal butane, and normal pentane are contrasted, and the relative merits of each are used as a basis of general discussion on layout of columns used in series.

Simple calculations on pressure distillation and stabilization are included in the discussion.

A. H. N.

198.* Viscosity and Constitution. G. Hugel. *Refiner*, August 1940, **19** (8), 294–296.—There is an important difference between chemical reactions and flow phenomena: in the chemical reaction only those collisions of molecules enter the picture which have been energetically activated, whilst in flow phenomena similar collisions have to be studied, but the molecules almost always carry a high energy level. It appears, then, that perhaps important characteristics of chemical constitution can have little or no bearing or influence on the viscosity of liquids.

This hypothesis is illustrated by plotting a mathematical function of viscosity of three liquids which are extremely different from a chemical point of view, dihydrodi-*iso*amyl anthracene, butandiol, and *n*-octane, and obtaining one curve for all three. The function was $\log \eta - a$ plotted against $T - b$, where η is viscosity, T is absolute temperature, and a and b are constants.

The simple exponential formula connecting viscosity and temperature (so-called Andrade Equation) is reviewed, and a linear relationship between the heat of activation of viscosity and the constant A is obtained for several substances which appear to yield a line common to all.

Dunstan's logarithmic law on the additivity of $\log \eta$ is studied in connection with the ketones which behave chemically as an associated liquid, and yet by viscometric studies according to Dunstan's law gives the same log increment for CH_2 as unassociated liquids. The behaviour of alcohols and glycol is studied. A. H. N.

199.* Elements of Vaporization and Condensation. Part VIII. Fractionator Design. R. L. Huntington. *Refiner*, September 1940, **19** (9), 354–358.—Laboratory fractionation of hydrocarbon gases is discussed both for low-temperature fractionation of natural gas and for ordinary high-temperature fractionation of heavier fractions. The difference in the boiling-point curves as obtained by the A.S.T.M. distillation apparatus and a true boiling-point column is briefly discussed and illustrated.

As an example, the fractionation of a binary mixture of simple hydrocarbons is studied. McCable–Thiele graphical method of evaluating the number of theoretical plates necessary is used to show the difference of high-pressure and atmospheric distillation processes, but the method is not sufficiently described for one interested in the "elements" of the subject. Mention is made of Sorel's algebraic method. Previous parts of the paper, however, have already dealt with the fundamental physical relationships between the variables of temperature, pressure and composition. [See *Ref.*, January–August, 1940, **19** (1–8).] A. H. N.

200.* Studies on the Separation of Paraffin Waxes. Part II. Phase Relationships. M. F. Sawyer, T. G. Hunter, and A. W. Nash. *J. Inst. Petrol.*, September 1940, **26** (203), 430.—Experimental results are reported for single- and multiple-stage separation of paraffin wax, after equilibrium has been attained between solid and liquid layers, and a theory is outlined for the ideal or infinite-stage fractional melting processes. A. H. N.

201.* Synthesis and Properties of Mono-Normal-Alkyl-Benzenes. Part I. G. Shen, T. Y. Ju, and C. E. Wood. *J. Inst. Petrol.*, October 1940, **26** (204), 475–487.—After critically examining the principal available methods for preparing *n*-alkylbenzenes, it is concluded that Friedel and Crafts' reaction is the best method for obtaining large quantities of hydrocarbons. Palladium is used for hydrogenating the alkyl aryl ketone which is the resultant of the Friedel–Crafts reaction. Both theoretical and experimental work is detailed, but Part I is devoted exclusively to theoretical considerations. A. H. N.

202.* Synthesis and Properties of Mono-Normal-Alkyl–Benzenes. Part II. T. Y. Ju, G. Shen, and C. E. Wood. *J. Inst. Petrol.*, November 1940, **26** (205), 514–531.—In this part of the paper the preparation, purification, and the properties are reported both for the intermediate ketones and for the corresponding hydrocarbons, in the synthesis of *n*-alkylbenzenes by Friedel and Crafts' reaction between benzene and corresponding acid chlorides to yield *n*-alkylphenyl ketones, which are reduced, by two methods, to the hydrocarbons. Large quantities have been prepared and their properties reported both as given in the literature and from the authors' experiments. A. H. N.

203.* Mechanism of Aromatization and Xylene Thermal Isomerization. A. F. Dobryanskii and F. Ya Saprykin. *Oil Gas J.*, 8.8.40, **39** (13), 48.—The yield of toluene and the structure of the xylene isomers formed by the pyrolysis of the pure xylenes at different temperatures have been examined. It has previously been shown that the pyrolysis of xylene at higher temperatures gives a higher yield of total xylenes and a greater preponderance of *m*-xylene over *o*-xylene than that at low temperatures. It is possible that this is due to isomerization of other xylenes to *m*-xylene at high temperatures. Pyrolysis of the three pure xlyene isomers was carried out in a porcelain tube at 700°, 730°, 750°, and 770° C., the yields of toluene from *o*-xylene increasing from 4·6% to 12·8% at increasing temperatures, that from *p*-xylene increasing from 2·1% to 13·3%, and that from *m*-xylene increasing from 1·7% to 15·4%. The xylene isomers formed (or unaltered) were separated, and it was found that *o*-xylene tended to form *m*-xylene in increasing quantities at high temperatures (5–38·2%), but relatively little *p*-xylene (traces to 10·5%). *p*-Xylene produces no *o*-xylene and only 1·3–7·7% *m*-xylene. *m*-Xylene is the most stable isomer and produces no *o*- or *p*-xylene on pyrolysis, the main products being toluol and condensable hydrocarbons.

From the data presented no conclusions can be reached as to whether *o*- and *p*-xylenes are subject to decomposition while in the *m*-xylene stage. It seems probable that decomposition and isomerization are simultaneous but independent reactions. C. L. G.

204. Cyclization of Dienynes. X. The Dodecahydrophenanthrone Obtained from Di*cyclo*Hexenylacetylene. C. S. Marvel, D. E. Pearson, and R. V. White. *J. Amer. chem. Soc.*, 1940, **62**, 2741–2743.—The ketone obtained by the cyclization of di*cyclo*hexenylacetylene with sulphuric acid has an α-, β-unsaturation as suggested by Linstead and Walpole. T. C. G. T.

205. Tertiary Naphthenic Acids. I. Synthesis of 1 : 2 : 3 : 3-Tetramethyl*cyclo*pentanecarboxylic Acid from Camphor. B. Shive, J. T. Horeczy, and H. L. Lochte. *J. Amer. chem. Soc.*, 1940, **62**, 2744–2746.—During research on the acidic constituents of petroleum, appreciable amounts of materials have been encountered which are not esterifiable by alcoholic hydrogen chloride. Since little is known regarding tertiary naphthenic acids, the work on such unesterifiable materials has been hampered.

1:2:3:3-Tetramethyl*cyclo*pentanecarboxylic acid has been synthesized from camphor and found to be different from the $C_{10}H_{18}O_2$ acid previously isolated. T. C. G. T.

206. Aromatic *cyclo*Dehydration. VII. Phenanthrene. C. K. Bradsher and R. W. Wert. *J. Amer. chem. Soc.*, 1940, **62**, 2806–2807.—Aromatic *cyclo*hydration or cyclization in which the elements of water are eliminated from a compound with the establishment of a new aromatic ring has been further developed and used in the synthesis of phenanthrene. T. C. G. T.

207. Catalytic Dehydrogenation of Alcohols in the Liquid Phase Using Ethylene as a Hydrogen Acceptor. W. Reeve and H. Adkins. *J. Amer. chem. Soc.*, 1940, **62**, 2874–2876.—Alcohols of four or more carbon atoms (*e.g.*, butyl, alcohol, or heptanol) may be dehydrogenated to aldehydes or ketones on the liquid phase. The reaction is carried out under 50–150 atms. ethylene, which latter accepts the released hydrogen. The reaction occurs at 280° C. The best catalyst is a mixture of copper, zinc, nickel, and barium chromites, but the reaction occurs with copper chromite alone. Barium seems to have a notable effect on retaining the activity of the catalyst, whilst zinc and nickel prevent polymerization of the aldehydes. T. C. G. T.

208. The Action of Elementary Fluorine on Organic Compounds. IX. The Vapour Phase Fluorination of Methane. E. H. Hadley and L. A. Bigelow. *J. Amer. chem. Soc.*, 1940, **62**, 3302–3303.—The vapour-phase fluorination of methane over a copper-gauze catalyst has been studied. Conditions were established when the proportion of products formed other than CF_4 could be increased to a maximum of 45 liquid vol.-% of the total condensate.

The products of fluorination contained all the fluoromethanes, C_2F_6, and C_3F_8. T. C. G. T.

209. Chlorofluoropropanes. E. J. McBee, A. L. Henne, H. B. Hass, and N. Elmore. *J. Amer. chem. Soc.*, 1940, **62**, 3340–3341.—$CH_3CF_2CH_3$, prepared from $CH_3C\ Cl_2CH_3$, has been chlorinated to produce several chlorofluoropropanes, including $CH_2Cl\ CF_2C\ Cl_3$ and $CH_2Cl\ CF_2CF_2Cl$, which were more pure than previously prepared compounds. New compounds were prepared by fluorinating $CH_2Cl\ CF_2C\ Cl_3$ to $CH_2Cl\ CF_2CF\ Cl_2$ and to $CH_2Cl\ CF_2CF_2Cl$ and by chlorinating $CH_2Cl\ CF_2CF_2Cl$ to $CH\ Cl_2CF_2CF_2Cl$ and to $C\ Cl_3CF_2CF_2Cl$. T. C. G. T.

Analysis and Testing.

210. Reflux Regulator and Head for Laboratory Rectifying Columns. F. D. Rossini and A. R. Glasgow, Jr. *Bur. Stand. J. Res. Wash.*, 1939, **23** (4), 509–513.—Although a number of reflux regulators and heads for laboratory rectifying columns has been described in the literature, none appears to fulfil all the special requirements arising in connection with the work of the A.P.I. Research Project 6 at the National Bureau of Standards.

These special requirements are that the device should (*a*) have a non-lubricated glass valve, (*b*) utilize a minimum of height above the column proper and permit the use of as long a rectifying section as possible, (*c*) provide for a substantially constant rate of removal of distillate for each setting of the valve, (*d*) provide for estimation of the throughput and reflux ratio, (*e*) provide space for a thermometric device, and (*f*) have a flexible metal partition for permitting distillation at pressures below or above the prevailing atmospheric pressure.

This paper describes an apparatus that possesses the above properties. D. L. S.

211.* Petroleum Solvents for Paints Compared by New Solvency Tests. S. S. Kurtz, Jr. *Nat. Petrol. News*, 24.7.40, **32** (30), R. 270–R.-273.—Increasing interest in the use of petroleum solvents in the paint industry has disclosed deficiencies in the nitrocellulose dilution ratio and the Kauri-gum butanol test. The viscosity of a dispersion of a resin in a petroleum solvent has been shown to be a sound and reliable guide to solvent power.

$$\text{Resin Solvency (R.S.)} = \frac{\text{viscosity of } X\% \text{ Resin in Standard Solvent}}{\text{viscosity of } X\% \text{ Resin in Solvent on Test}}.$$

The preparation of resin solutions is a time-consuming operation, and time is saved by the use of a graphical method based on a correlation between R.S. and specific gravity, refractive index at 20° C., and the 50% A.S.T.M. end-point. The tests are reliable only for two-fold systems, simple solutions of one resin in a spirit. Current investigations are expected to extend the application of the tests to systems in which a third principle is present. H. G.

212. Significance of Doctor Test for Gasoline Found Negligible. J. Happel and S. P. Cauley. *Nat. Petrol. News*, 7.8.40, **32** (32), R. 284–R. 290; *Refiner*, June 1940, **19** (6), 205–208.—The paper comprises a critical discussion of the significance of the doctor test as a gasoline specification item from the points of view of both consumer and refiner. Data are presented to show that although from the consumer's point of view the test is used to control odour, in many cases the two factors are unreliably related. The practical limit of mercaptan concentration, when odour only is the criterion, is usually greater than that demanded by the sensitivity of the test. From the refiner's point of view it is suggested that the gain from the standpoint of odour is often outweighed by a quality loss in the shape of a reduced octane number after treatment. It is argued that in the interests of all concerned the mercaptan concentration of a finished gasoline should be controlled by considerations of economics, and should not be dependent on an arbitrary test. It is concluded that a greater consciousness of properties of more significance than that of odour and a growing realization of the lack of its significance should combine to eliminate the doctor test from gasoline specifications. H. G.

213.* Reduction Chart for the Stormer Viscometer. H. T. Byck. *Oil Gas J.*, 8.2.40, **38** (39), 40.—In the measurement of the viscosities of drilling muds by the Stormer instrument the rotor speed is fixed at 600 r.p.m. and the driving weight necessary to produce this speed is determined. This driving weight is converted into absolute viscosity by a calibration chart obtained by measuring a series of glycerine solutions of known viscosity in the same instrument. The rotor speed is adjusted by variation of the driving weight until 100 revolutions take place in exactly 10 sec. In practice this is tedious, and a means of arriving at the value for 10 sec. by interpolation from values on either side of the correct one has been devised. Provided the times for 100 revolutions are in the one case not less than 8 sec. and in the other not more than 12 sec., the curve driving weight against time is a straight line. A chart on this basis is provided and its application illustrated. R. J. E.

214.* Vapour Bomb Connection Saves Pressure Gauges. D. Attaway. *Oil Gas J.*, 1.8.40, **39** (12), 41.—In carrying out the Reid Vapour-Pressure Test (A.S.T.M. D. 323–37 T) the air-chamber and gauge are subjected to numerous and vigorous shakings, resulting in damage to the spring-gauge mechanism and necessitating frequent recalibration and repair. A description is given of a needle-valve and collar which may be inserted between the air-chamber and gauge, thus allowing the gauge to be removed before shaking. Check tests against a standard assembly agree to within 0·1 lb. C. L. G.

215.* Calculating Gasoline Octane Rating from Gravity and A.S.T.M. Distillation. R. B. Cox. *Refiner*, February 1940, **19** (2), 31–36.—The paper presents methods of calculating the octane rating of any gasoline by using only its A.P.I. gravity and A.S.T.M. distillation. All gasoline samples are subjected to at least these two tests, even in control work, so that the calculation proposed here requires no tests other than those that are made in any case. This obviates the objection that has been raised to other methods of octane number predetermination. Moreover, these two basic tests are simple, reproducible, accurate, require no complicated apparatus, and may be determined by one operator in a total elapsed time of 35 min. In fact, with a little experience one operator can run three distillations simultaneously.

The basis of this correlation is the fact that when the boiling point is plotted against gravity for 180 hydrocarbons generally found in straight-run and cracked gasolines, the types fall in fairly well-defined areas, with the saturated straight-chain paraffins forming the upper boundary of the top region, and the aromatics the lower region. The rest of the development of the charts and arguments are empirical.

It is pointed out that the charts are naturally no more accurate than the test data. It is absolutely imperative that the distillation be run exactly to A.S.T.M. specifications. Also, the distillation must be run immediately after the gravity is determined. Allowing the sample to stand for even a few minutes after the gravity is taken before running the distillation is a common source of error, owing to evaporation at room temperature of the lighter fractions. Such evaporation changes the distillation curve, particularly the 10% point. A. H. N.

216.* Determination of Tetraethyl Lead in Gasoline. G. Calingaert and C. M. Gambrill. *Refiner*, February 1940, **19** (2), 58–60.—Tetraethyl lead is determined quantitatively by refluxing the gasoline with concentrated hydrochloric acid, extracting the lead chloride with water, and determining the lead found by any of the standard methods.

A table indicates that the new method yields results with a mean error of only − 0·012 ml. of tetraethyl lead/gall. of gasoline, as against − 0·054 for the bromination method, the improvement being particularly noticeable in the case of cracked gasolines. Furthermore, the precision of the new method is greater than for the bromine method, the upper limit for the variation between laboratories being 0·050 ml. of tetraethyl lead/gall. of gasoline by the new method, as compared with 0·075 by the bromine method. A. H. N.

217.* New Firing Time Test. J. DeMent. *Refiner*, August 1940, **19** (8), 282–284.—It was noted that many organic inflammable liquids, when ignited, burned with a time characteristic for each sample. Further, when a standard procedure was followed, the flames, deposits, residues, colours, types, and heats of flames were also characteristics of the liquid burning. Thus a new test is outlined where a measured quantity of liquid is slowly poured into a burner, ignited, and the time it takes to burn completely is noted with a stop-watch.

Experimental procedure and standardized apparatus and technique are detailed. A table gives characteristics for various liquids, the firing time period varying from 39·1 sec. for a sample of methanol, to 95 sec. for a sample of gasoline under standardized conditions. To illustrate the repeatability and precision of the test for liquids requiring rapid technique 20 test results are reported for a sample of methanol, the average being 45·0 sec., with a minimum value of 41·6 sec., and a maximum of 47·2 sec.

A modified test is suggested for liquids and solids which do not burn easily, which test uses a solvent for the liquid or solid being studied.

A scheme of fire time determination in which various properties of the flame are noted both qualitatively and quantitatively is detailed, and a list of possible applications and modifications of the test ends the paper. A. H. N.

Motor Fuels.

218.* High Anti-Knock "Safety" Aviation Fuel. M. G. van Voohis. *Refiner*, January 1940, **19** (1), 19–21.—It seems that the solution of the problem of aviation fuels and their better utilization in the modern multi-cylinder engine is mostly viewed in relation to replacing the carburettor with a device for direct injection.

A saving of 5% gasoline is secured by the use of direct injection of the fuel, this being quite appreciable in the field of aviation mechanics. Another advantage is in testing work where the multi-cylinder engine can be easily replaced by a single-cylinder engine, lowering the cost and simplifying the test procedure.

The direct-injection motor is manufactured extensively in Germany, and its use will no doubt result in the utilization of a far less volatile fuel than gasoline, resulting in the wide adaptation of this type of motor. The motor functions at once with any setting of the injection pump, which is of paramount value in airplane flights. From a military point of view, it can be mentioned that this motor allows the pilot to approach his objective in gliding formation absolutely silently once he has attained a predetermined height. It is also important in similar type planes to point out the great fire hazard when using very volatile fuels, their hazardous storage in the supply tanks, and the use of complicated feed systems. The use of "internal cooling" is more easily obtained, since regulation of the injection is easier and safer when controlling the single air inlet.

The idea of using safety fuels to increase the safety of airplane operations is so important as to necessitate the most thorough scientific and mechanical investigations. It is of course understood that such safety fuel will be a highly efficient and otherwise scientifically refined product showing no lowering of engine efficiency. Tests and aspects of safety in using these fuels with low fire hazards are discussed briefly.

A. H. N.

219. Patent on Motor Fuel. A. R. Goldsby. U.S.P. 2,221,165, 12.11.40. Appl. 15.10.40. Conversion of lower-molecular-weight hydrocarbons into higher-molecular-weight hydrocarbons. Straight-chain and branched-chain paraffinic hydrocarbons are subjected to the action of chromic acid and a metallic halide at a sufficiently high temperature to effect a substantial amount of dehydrogenation and alkylation to produce anti-knock hydrocarbons within the motor-fuel boiling range. H. B. M.

See also Abstracts Nos. 212 and 215.

Diesel, Gas and Fuel Oils.

220. Patents on Gas, Diesel and Fuel Oil. H. Rupp. E.P. 528,093, 22.10.40. Appl. 27.4.39. Preparation of a colloidal fuel in which the solid particles are in very stable suspension in the liquid fuel. The suspension, or first the liquid fuel and then the suspension of coal dust, is maintained under vacuum and the gas injected by means of an atomizer. Thereafter the suspension is allowed to return to normal pressure.

D. E. Badertscher and M. S. Altamura. U.S.P. 2,218,447, 15.10.40. Appl. 15.12.38. Preparation of a diesel fuel oil in which is incorporated a small proportion of the aliphatic thiamine obtained by reacting a primary aliphatic amine with sulphur dichloride. H. B. M.

Lubricants and Lubrication.

221.* Non-Fluid Lubrication. Part III. S. Kyropoulos. *Refiner*, March 1940, **19** (3), 85–92.—The various physico-chemical factors are discussed, determining the behaviour of bearing metals in non-fluid lubrication under the influence of local high temperatures and chemical reactions with the lubricant which are liable to occur. Journal polish is in general to be considered both as a mechanical protection against excessive cold work by greater smoothness, and as a physico-chemical protection by a protective anti-welding oxide layer. The coefficient of kinetic friction will be that of the oxidized metal.

A new theory of bearing metals is presented, explaining the mechanical part played by the various constituents of a composite bearing metal. The load-carrying capacity of a simple structure is calculated by stress analysis. The theory, derived from observation, provides a simple rule for the selection of bearing alloy components and indicates general rules for the relative particle size.

In a well-composed bearing metal for high speed and temperature it is the soft component which directly carries the load, because of its higher thermal expansion. Due to the part played by volume expansion, the relative particle size of the constituents determines frictional and seizure properties. The number of metals with outstanding thermal expansion is limited to Cd, Pb, Sn, Al, and Ag. Their melting points are upper limits of "high-spot" temperatures, and consequently limit possible physico-chemical reactions—*e.g.*, diffusion-seizure with the journal. Cd, Pb, and Sn are, for such reasons, the easiest to handle. Because of their low yield and melting points, their friction must necessarily be low.

Fatigue resistance and mechanical strength increase, in a general way, with the melting point. For the same reason the work of deformation increases, as does the kinetic friction—comparing *e.g.* Sn and Al—because there is no kinetic friction without cold work and wear. It is for this reason that any pure high-melting metal must tend to " grip " any journal under certain conditions of clearance and temperature, no matter whether this " gripping " is accompanied by true seizure (welding, diffusion) or not.

In connection with fatigue phenomena the physical fundamentals of crystal strength and Griffith's theory of rupture are briefly discussed with regard to possible effects of differential expansion on the intercrystalline stresses. The problem of intracrystalline reinforcement is briefly discussed, and a possible attack of the problem of reinforcement against fatigue from the lubrication angle is suggested. A. H. N.

222.* Effect of Engine Metals on the Deterioration of Motor Oil. B. F. Downing, G. E. Holbrook, and J. H. Fuller. *Oil Gas J.*, 15.6.39, **38** (5), 70.—The catalytic oxidation effect of various metals on transformer oils and lubricating oils has been studied by laboratory tests (bibliography given), but no information has been published on the effect of engine metals, as determined in full-scale engine tests. To obtain these data tests were carried out on a 6-cylinder 1936 Chevrolet engine, the effect of the different metals present being separated by making tests before and after selected surfaces had been protectively lacquered by the resinous film from lubricating oil at high crankcase temperatures. The effect of iron was maximized by cleaning the engine in 5% trisodium phosphate solution, and that of bearing metals by using new bearings. Oil samples were drawn from the sump after 10 min., 1 hr., 3 hrs., and 4 hrs., and analysed for (1) total sludge (A.S.T.M. naphtha insoluble), (2) asphaltenes (total sludge soluble in chloroform), (3) ash, and (4) iron. From the total sludge, ash is deducted, giving hydrocarbon sludge, allowance being made in determining the final sludge figure for the sludge in the samples taken. It is shown that the total catalytic effect of crankcase iron is much greater than that of Cu–Pb, Ag–Cd, and Babbitt metal in connecting rod bearings. Of the bearing metals Cu–Pb is the most active, with Babbitt and Ag–Cd following in that order. The addition of 0·5% of phosphorus additive (80% tricresyl phosphate and 20% tributyl phosphite) reduced total hydrocarbon sludge by 51·5/83·1% and connecting-rod bearing wear by 52·8/96·5%, iron being still the predominating influence, and gave uniformly lower oil deterioration. It is concluded that approximately two-thirds of the oil oxidation and deterioration in a clean or new engine is due to catalysis by engine metals. The effect of the phosphorus additive is attributed to retardation of solution of metal in the oil and inhibition of the catalytic effect of the metal on the oxidation of the oil. This is particularly the case with new copper lead bearings until they acquire a lacquered surface.

C. L. G.

223.* Piston Deposits, Ring Sticking, Varnishing and Ring Clogging. W. A. Gruse and C. J. Livingstone. *J. Inst. Petrol.*, September 1940, **26** (203), 413–429.—It is found that engine-varnishing, ring-sticking, and oil-ring clogging are more or less closely related. Oxidation of oil, chiefly in the crankcase, to unstable products, followed by the decomposing of these products at hot points in the engine, play an important part in all three. Cooling of crankcase oil is strongly advocated.

A. H. N.

224. Patents on Lubricants and Lubrication. E. A. Evans and C. C. Wakefield & Co., Ltd. E.P. 527,695, 15.10.40. Appl. 29.12.38. Improvements in extreme pressure lubricants comprising a mineral oil and a chlorine-containing naphthalene derivative. In this case the sole chlorine-containing naphthalene derivative consists of a minor proportion of naphthalene tetrachloride dissolved or suspended in the lubricating oil. The resultant product has a high film-rupture strength, and is accordingly particularly useful for the lubrication of moving parts in internal-combustion engines.

Armour & Co. E.P. 528,096, 22.10.40. Appl. 27.4.39. Improvements in the preparation of extreme-pressure lubricants composed of a lubricating agent to which has been added an alkyl thioamide to impart the desired characteristics.

Hilliard Corp. E.P. 529,209, 15.11.40. Appl. 24.5.39. Apparatus for purifying used lubricating oils. A filter is arranged to discharge the filtered oil into a vapourizing chamber containing a heated member formed with a number of comparatively shallow channels combining to form a tortuous path along which the oil must flow in a relatively thin stream before escaping from the chamber. The heated member is so arranged that the oil is heated prior to its passage through the filter, and the said chamber is connected to a source of suction which facilitates liberation of contaminating volatile constituents from the heated oil.

R. Rosen. U.S.P. 2,216,752, 8.10.40. Appl. 3.4.37. Preparation of a petroleum lubricating oil suitable for use at high temperatures and containing as an oxidation inhibitor a small amount of an oil-soluble compound with an aromatic nucleus.

B. H. Lincoln and W. L. Steiner. U.S.P. 2,217,173, 8.10.40. Appl. 19.2.36. Preparation of a high-film-strength lubricating oil consisting of a major proportion of hydrocarbon oil, 0·5–10% by weight of an aliphatic ester and 0·001–1% by weight of pinene.

W. G. Horsch. U.S.P. 2,217,368, 8.10.40. Appl. 12.3.38. Method of removing low-temperature sludge from dielectric mineral-oil compositions by adding thereto a substance selected from the group consisting of diphenyl and diphenyloxide.

F. Rostler and V. Mehner. U.S.P. 2,217,919, 15.10.40. Appl. 25.5.39. Production of a viscous mixture of unsaturated hydrocarbons containing 90% carbon and 10% hydrogen. The composition boils between 150° and 360° C., at 12 mm. Hg absolute pressure.

B. M. Lincoln, W. L. Steiner, and J. D. Byrkit. U.S.P. 2,218,132, 15.10.40. Appl. 2.5.38. Preparation of a lubricating-oil composition consisting of a major proportion of a hydrocarbon oil and from 0·05 to 25% of a sulphurized substantially pure olefin.

E. W. Fuller. U.S.P. 2,218,283, 15.10.40. Appl. 8.3.39. Preparation of a stabilized mineral-oil composition consisting of a highly refined viscous oil and a minor proportion of a mercapto-benzo-thiazole.

J. G. McNab and W. T. Watkins. U.S.P. 2,218,618, 22.10.40. Appl. 25.2.39. Preparation of an improved lubricant consisting of a mineral lubricating oil and a polyvalent metal soap of substantially non-distillable organic acids.

C. M. Loane and B. H. Shoemaker. U.S.P. 2,218,918, 22.10.40. Appl. 31.8.36. Preparation of a lubricating oil to which has been added a substantial amount of an alkyl thiocyanate having 8 to 14 carbon atoms in the alkyl radical.

F. R. Moser and M. C. Tuyn. U.S.P. 2,219,164, 22.10.40. Appl. 16.3.36. Preparation of an extreme-pressure lubricant consisting of a substantial quantity of mineral oil and a small proportion of a compound of the formula $R(SO_2X)m$, wherein R is an aliphatic radical, m is one or two, and X is a halogen.

R. C. Moran. U.S.P. 2,220,941, 12.11.40. Appl. 4.2.39. Preparation of a stabilized mineral-oil composition consisting of a viscous mineral-oil fraction having incorporated therein a minor proportion of a dimercaptyl diethyl ether in an amount sufficient to inhibit the deleterious effect of oxidation on the oil.

C. M. Loane and B. H. Shoemaker. U.S.P. 2,220,970, 12.11.40. Appl. 18.12.39. Preparation of a lubricant containing about 0·05–10% of an organic compound selected from the group consisting of (1) pseudo-thiourea derivatives and (2) the condensation products of thioureas and mustard oil.

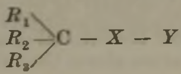
C. T. Anne, H. I. Wilson, and W. C. Patterson. U.S.P. 2,221,161, 12.11.40. Appl. 25.8.38. Method of preparation of a lubricating oil suitable for use as cylinder stock, substantially free from asphaltic and acidic constituents, and having a desirable green cast, from reduced naphthene base crude oil.

H. V. Ashburn and W. G. Alsop. U.S.P. 2,221,162, 12.11.40. Appl. 6.8.38. Preparation of an oil for lubricating the bearings and cylinders of internal-combustion engines. The composition comprises a mineral lubricating oil within the motor-oil viscosity range, 0·01–2·0% of a phosphatide to inhibit bearing corrosion and lacquer formation, and less than 1% of an emulsion preventing agent.

P. J. Wiezevich. U.S.P. 2,223,473, 3.12.40. Appl. 16.10.37. Preparation of a lubricating composition consisting of a petroleum lubricating oil and 0·5–30% of a licanic metal soap soluble in the lubricating oil.

B. H. Lincoln, W. L. Steiner, and G. D. Byrkit. U.S.P. 2,223,766, 3.12.40. Appl. 2.2.40. Preparation of a lubricant consisting of a hydrocarbon oil intimately admixed with 1–10% by weight of a halogenated, aromatic ketone.

N. E. Peery. U.S.P. 2,223,793, 3.12.40. Appl. 14.11.38. Preparation of a lubricating composition consisting of a mineral oil and an amount sufficient to impart extreme pressure properties of a compound of the structure



$$\begin{matrix} R_1 \searrow \\ R_2 \rightarrow \\ R_3 \nearrow \end{matrix} C - X - Y$$

R_1, R_2, and R_3 represent aryl radicals, X is an element selected from the group consisting of oxygen and sulphur and Y is a phosphorous halide radical containing 2 halide atoms attached to the phosphorous.

S. Musher. U.S.P. 2,223,941, 3.12.40. Appl. 29.11.39. Method of improving lubricating oils by decreasing sludge formation and tendency to cause corrosion of metal surfaces. The oil is reacted with a relatively small amount of combination of lethicin and a phosphite ester at a temperature above 350° F. H. B. M.

Asphalt and Bitumen.

225.* Continuous Automatic Blending for Cutback Asphalts. Anon. *Refiner*, August 1940, **19** (8), 261–263.—A description is given of a continuous automatic blending plant the capacity of which is 500 brl./hr. for blends which may range from 95% asphalt and 5% solvents to 50% of each. A single adjustment, maintaining the percentage of solvent in the final product, is sufficient to give any of twelve cutback asphalts continuously and without material deviation.

The plant is essentially composed of two units : a mixing unit and a solvent metering unit which is, in effect, a displacement meter which measures the quantity of solvent introduced into the asphalt upstream from the blending and mixing meter. An adjusting device on the solvent metering unit is set to adjust the ratio of solvent to the finished cutback. The operation and method of adjustment of the plant are detailed.

As solvent and asphalt are mixed under pressure in a closed system, foaming and loss of light fractions are avoided. When the correct proportions have been ascertained, and the plant set to deliver the required quantity of solvent, there appears to be but a slight deviation from specifications at the delivery end of the pipe line. A table giving data of a typical cutback illustrates this statement. A. H. N.

226. Road Oil Sales Increase 5% in 1939. R. R. Sayers. U.S. Dept. of the Interior, Bur. Mines, Mineral Market Reports, M.M.S. No. 851.—The report gives figures for refinery sales of road oil in the United States during 1939 and indicates an increase of 5% over the total for 1938. This was largely due to increased construction of oil-treated macadam, gravel, and sand-clay highways. Nevertheless, owing to lower prices, the value at the refineries of sales of road oil decreased by 11% during this period. Of the 8,108,400 brl. of road oil sold in 1939, only 293,100 brl. were made from foreign petroleum, imported chiefly from Venezuela and Mexico.

In 1939, 7,868,000 brl. of road oil were produced in United States refineries, as compared with 7,788,000 brl. in 1938. This output was increased by 1,222,700 brl. of other products, chiefly fuel oil, transferred to road-oil stocks. Thus, stocks of road oil and transferred oil increased by 22,000 brl. during 1939, as compared with an increase of 13,000 brl. during 1938. H. B. M.

227. Patents on Asphalt and Bitumen. K. Dammann. E.P. 527,816, 16.10.40. Appl. 21.3.39. Production of a bituminous road surface which, owing to the presence of numerous fragments of stone chippings, is extraordinarily rough, but in which the chippings are so firmly embedded that they cannot be torn out by traffic. The surface is effected by consolidating the chippings simultaneously with a sand-like asphalt mass or bituminous plastic layer by means of a consolidating implement, the operating face of which is fitted with a yielding layer of rubber. Thus the chippings become firmly anchored in the asphalt mass and at the same time project above its surface.

N.V. De Bataafsche Petroleum Maatschippij. E.P. 528,653, 4.11.40. Appl. 11.5.39. Manufacture of asphalt from products containing both asphalt and paraffin wax, and which have been obtained in the straight-run distillation of mineral oil. The products are first heated above the temperature at which disruption of dissolved molecular paraffin-wax aggregates occurs, subsequently cooled and deasphaltized within about 1 hr. of cooling below the critical disruption temperature, with the aid of said hydrocarbons at a temperature above that at which the paraffin wax begins to settle out.

A. B. Brown, F. C. Croxton and B. Ginsberg. U.S.P. 2,217,727, 15.10.40. Appl. 9.6.38. Method of obtaining high-melting point asphalt and bright stock from mixed-base crude residua.

A. P. Anderson. U.S.P. 2,223,776, 3.12.40. Appl. 21.10.38. Manufacture of asphalt having a high viscosity at elevated temperatures and good ductility at low temperatures. An asphaltic bituminous material is blended with a hydrocarbon material having a viscosity not less than 600 sec. (Saybolt) at 275° F., and a viscosity-gravity constant above 0·870. The hydrocarbon material is at least 95% soluble in 86° β naphtha. H. B. M.

Special Products.

228. Naphthenic Acids in Specialty Formulas. B. Levitt. *Chem. Industr.*, 1940, **47** (2), 166.—Although naphthenic acids derived from petroleum sludges have not as yet been completely isolated and identified, they have already found extensive use in the manufacture of soaps, paints, fungicides, etc. Based on a yearly world production of 200 million tons of petroleum and an average of 0·1% content of naphthenic acids, there is potentially available for recovery 200,000 tons of acids per year. In the U.S.A. the greater part of the naphthenic acids used are converted into metallic naphthenates for use as paint dryers, whilst in many European countries where fats are scarce these acids are used to a certain extent in the manufacture of soap. This article briefly describes the methods of recovery and properties of naphthenic acids, together with the preparation and uses of the metallic naphthenates. B. M. H. T.

229.* Petroleum Sulphonic Acids. S. Pilat and J. Sereda. *Refiner*, February 1940, **19** (2), 50–52.—From the products of interaction of sulphuric acid with mineral oils, sulphur derivatives of phenols occurring in crude oils were isolated for the first time. After splitting off the sulpho group from the phenolsulphonic acids, phenols were obtained having a molecular weight of about 260 and an empirical formula $C_{17}H_{26}OH$.

It was shown that the colour reaction of iron petroleum-sulphonates is due to the presence of petroleum phenolsulphonic acids for which it is characteristic.

Iron salts of petroleum phenolsulphonic acids are soluble in organic solvents, producing an intensive blue or green colour. Water-soluble mineral salts, in solid form or in the form of aqueous solutions, bring about a disappearance of the colour. Likewise, the effect of temperature or of traces of water is to bring about a disappearance of the colour or its reappearance.

Some changes observed in the iron salts of petroleum phenolsulphonic acids can be explained by the action of the hydroxyl group in the reaction with ferric chloride and by the phenomenon of tautomerism and desmotropism. A. H. N.

230. Waxes from Fatty Acids. German Conversion Process. Anon. *Chem. Tr. J.*, 18.8.39, **105**, 140.—The production of materials similar to wax or petroleum jelly by heating distillation residues of fatty acids (*e.g.*, obtained from fatty oils, or oxidation of paraffin hydrocarbons) in the presence of catalysts, followed by reduction with hydrogen, is described by I.G. Farbenindustrie A.-G. in E.P. 506,678 of 1938. The catalyst used—*e.g.*, metallic iron or nickel or manganese oxide—splits off CO_2 at sufficiently high temperatures—*e.g.*, 200° C.—the carboxylic acids condensing to give ketones. Filtration or sedimentation removes the catalyst. Reduction with hydrogen converts carbonyl and carboxyl groups into hydroxyl and/or methylene or methyl groups, any double linkages being thus saturated. Hydrogenation is carried out in the presence of catalysts and preferably at 160–200 atmospheres pressure and 200–400° C. The resulting products are pale, frequently colourless, similar to wax or petroleum jelly, consisting mainly of high-molecular-weight alcohols and/or hydrocarbons. They are suitable as medicinal or veterinary salves, or for the protection of metals against industrial or other gases. A typical product from crude fatty acids obtained from Fischer–Tropsch paraffin wax was similar to petroleum jelly, and smeared well, exerting no irritant effect on the skin. It had an acid value of 0, saponification value 0, and melting point 61° C. C. L. G.

231. Synthetic Rubbers. A Review of their Compositions, Properties, and Uses. L. A. Wood. U.S. Nat. Bur. of Standards Circ. C. 427, 1940.—A detailed discussion, with bibliography, is presented of the methods of production, raw materials used, chemical reactions, general conditions of polymerization or condensation, and properties of twenty-nine commercial varieties of synthetic rubber. These are classified as : (1) chloroprene polymers (*e.g.*, Neoprene) ; (2) butadiene polymers and co-polymers (*e.g.*, Buna) ; (3) organic polysulphides (*e.g.*, Thiokol) ; (4) *iso*butene polymers (*e.g.*, Vistanex) ; (5) plasticized vinyl chloride polymers (*e.g.*, Koroseal), and (6) methyl rubber. General applications, their comparison with natural rubber, quantities produced, and cost of production of some of the more important types are outlined.

C. L. G.

232. Patents on Special Products. Standard Oil Development Corp. E.P. 528,792, 7.11.40. Appl. 24.4.39. Preparation of improved viscous liquids for use in shock absorbers, dash-pots, vibration dampeners, etc. The composition has a petroleum-oil base which blends with a minor proportion of a viscosity building oil to form a hydraulic medium.

E.I. Du Pont de Nemours. E.P. 529,311, 19.11.40. Appl. 15.5.39. Apparatus for the recovery by solvent extraction of extractable materials, particularly oils, fats, and waxes, from solid materials containing these substances. The apparatus comprises a long extractor tube inclined to the horizontal and connected at its lower end with the lower part of a short riser tube, means for delivering solvent to the upper part of the short tube, means for discharging lixivium from the upper part of the long tube, and means for moving solid material downwards through the long tube and upwards through the short tube.

Compagnie Française de Raffinage. E.P. 529,429, 20.11.40. Appl. 31.5.39. Preparation of active wave-stilling oils by partly saponifying a natural glyceride oil having an iodine number not less than 120, to produce a mixture of the fatty acids thereof with unchanged oil in which the fatty acids constitute between 30% and 70% by weight of the mixture.

W. G. Horsch. U.S.P. 2,221,380, 12.11.40. Appl. 25.1.38. Preparation of an oil composition for use in turbines, transformers, etc., which is capable of cleaning and maintaining clean the passages of such equipment. The composition consists of a homogeneous mixture of a mineral oil and a compound selected from the group acetophenone and benzophenone. H. B. M.

Detonation and Engines.

233.* Factors Involved in Lead Susceptibility. A. W. Trusty. *Refiner*, April 1940, **19** (4), 93–96.—The detonation theory, the spontaneous theory, and the peroxide theory of knocking are explained. The rate of rise of pressure cannot be the sole factor determining the tendency of a fuel to knock. The rate of pressure rise (1) increases with the molecular weight of the normal paraffin hydrocarbons; (2) varies inversely with the number of methyl groups attached to an aromatic nucleus; (3) is approximately the same for hydrocarbons of widely varying anti-knock properties, for example, benzene and *n*-octane, toluene and *n*-heptane, xylene and *n*-hexane; (4) about the same for the higher alcohols (high anti-knock compounds) as for the corresponding paraffin hydrocarbons (low anti-knock compounds); and (5) is very rapid for ethyl ether.

The lead susceptibilities of pure hydrocarbons are found to be: (1) the lower-boiling paraffins and naphthene, or those hydrocarbons with the highest anti-knock values are most susceptible. There is a marked difference between the light and heavy hydrocarbons. (2) The aromatics are fairly susceptible to tetraethyl lead Aromatics with a saturated side-chain are more reactive to lead than when the saturated side-chain is replaced with an unsaturated side-chain. (3) The unsaturated hydrocarbons, on the other hand, show much less variation in lead susceptibility, regardless of their original octane number.

As the double bond progressively moves towards the centre of the molecule, the effectiveness of lead in the compound increases. With a number of unsaturated hydrocarbons, lead has a " negative " effect—that is, the octane number is actually lowered by the addition of tetraethyl lead.

Acid treatment of cracked gasolines removes sulphur compounds and certain of the unsaturated hydrocarbon constituents. The removal of the unsaturates results, in general, in a lowering of the octane number, but by the removal of these same hydrocarbons, the lead susceptibility is raised.

Natural gasoline has in general the highest susceptibility to tetraethyl lead of all commercial blending stocks.

Low-sulphur, paraffin base, straight-run gasolines have the next highest effectiveness to the addition of lead.

High-sulphur, naphthene base, straight-run gasolines are considerably less reactive to lead blending than the low-sulphur, paraffin-base gasolines.

Cracked gasolines have lower lead susceptibilities than straight-run products—the degree depending on the sulphur content and the extent of cracking to which the stock was subjected. A. H. N.

234.* Octane Number. W. W. Scheumann. *Refiner*, April 1940, **19** (4), 102.—The paper is non-technical in treatment, giving a review of the concept and determination of octane numbers, the limitations of the method, and a general as well as specific warning against its misuse.

The discussion further points out that the octane number in any of its modifications is not a true measure of intrinsic anti-knock quality of fuels. It can never truly correlate with all types of car performance on the road, because such performance depends on engine characteristics and type of operations as much as on fuel characteristics. The oil companies have, therefore, greatly over-emphasized the value of the octane number, and have misused it in many ways.

Anti-knock quality in motor fuel is of definite economic value to the consumer. However, present engines do not use this quality most effectively, and research indicates that engines could be improved in this respect. In the past such improvements were not always made, because the refiner lavishly supplied additional anti-knock quality in his fuel to make up for such shortcomings. Only time will tell how much longer he can continue this practice. A. H. N.

235.* Knocking Octanes. C. H. Van Hartesveldt and H. W. Field. *Refiner*, June 1940, **19** (6), 209–214. *Paper Presented before American Petroleum Institute.*—In this paper a comparison is presented of the value of improved automobile performance as observed by the " average driver," with the incremental costs of producing gasolines of successively higher octane numbers.

The automobile-performance observations have been made in car models now being operated by the public, rather than in engines specifically designed for extremely high octane gasolines.

Further, it is notable, in view of many recent controversies, that " road octanes " are used throughout the discussion, even though it is admitted that the road-octane method leaves something to be desired in the way of reproducibility from car to car.

The improvement in the performance of present-day motor-cars, as a result of wide-range increases in gasoline octane numbers, is shown to be minor relative to the increase in refinery cost of effecting these octane increases as reflected in potential reduction of the profit returns on investments in the petroleum industry.

A note of caution is struck, lest refiners let competitive enthusiasm undermine the financial stability of the industry instead of proceeding on a sane and logical course of developments for the future. A. H. N.

236.* Behaviour of Gasoline-Coal Fuel in Spark-Ignition Engines. J. E. Hedrick. *Refiner*, August 1940, **19** (8), 285–286.—Data are presented on engine performance where a gasoline–coal suspension was used, and the results are compared with those obtained when the same gasoline was used alone. The gasoline had a specific gravity of 0·7234 at 60° F. and the mixture had 0·7332 specific gravity at 60° F., containing 2% by weight of coal of a particle size ranging from 1 to 74 microns. The engine had four cylinders and its speed was rated at 1200 r.p.m. Other data are included.

Although no appreciable detonation developed with gasoline even at maximum load, the use of the mixture gave a prominent detonation at the same load. The engine developed less power, due probably to detonation, when coal suspension was used. Further, on a constant load test, the engine was unsteady, alternately developing severe knock and then running smoothly for a while. The exhaust was clean, and no smoke developed with the use of coal, but the tests were too short to enable a study to be made on the effects on the cylinders. Coke in loose flakes was deposited on the entire combustion chamber. The lubricating oil suffered badly, and tar was formed on the valve stems and drained into the crankcase. A. H. N.

237.* Mileage Marathon Develops Interesting Data. R. J. Greenshields and L. E. Hebl. *Oil Gas J.*, 1.8.40, **39** (12), 33.—Useful engineering data, mainly on methods of increasing mileage, have been obtained as a result of a mileage contest on a definite quantity of fuel. Lighter cars are handicapped on a theoretical basis, using data on published friction values based on actual road measurements, on wind resistance, mechanical efficiency, and a number of other assumptions. Methods of increasing mileage included: adjustment of spark timing and carburettors, inflating tyres to 50 lb. per sq. in., adjusting wheel alignment, use of exhaust heated differential housing and insulated crankcase and transmission (based on article by Hebl & Rendel, *N.P.N.* 24.7.35, p. 36), shielding radiators to give maximum engine temperatures and disconnecting fans, water-pumps, and generators. A new driving technique was also successfully used, in which the engine was accelerated at full throttle from 16 to 37 m.p.h., and then was coasted to 16 m.p.h. with the ignition off. C. L. G.

238.* Clement–Pigneguy Knock Indicator. D. M. Clement and P. G. Pigneguy. *J. Inst. Petrol.*, November 1940, **26** (205), 489–514.—An indicator is described which is unaffected by combustion pressure, and which allows testing to be carried out over the entire octane scale without alteration of the original setting. A. H. N.

239. Patent on Detonation and Engines. F. M. Pyzel. U.S.P. 2,218,137, 15.10.40. Appl. 2.8.37. Subjecting a gasoline-type motor fuel containing 5–60% methyl *iso*butyl carbinol to combustion in a spark ignition engine as an anti-knock fuel. H. B. M.

Economics and Statistics.

240. Market for Liquefied Petroleum Gases Expands a Third in 1939. R. R. Sayers. *U.S. Dept. of the Interior, Bur. Mines, Mineral Market Reports*, M.M.S. 842.—Figures given in this report indicate a definite increase in sales of liquefied petroleum gases during 1939 as compared with 1938. With the exception of chemical manufacturing, the increase is reflected in all important uses of these gases. Sales of " bottled gas " employed largely in kitchen equipment in rural districts situated beyond the city gas mains rose by more than 50% in 1939. Gas companies using liquefied petroleum gases for enriching manufactured gas or for direct distribution increased their purchases by nearly 37% during the year. Also industrial demand which had shown a decline in 1938 as compared with 1937 had an outstanding increase of 59% in 1939.

During 1938 deliveries of liquefied petroleum gases were divided in approximately equal proportions between propane, butane, and propane–butane mixtures. During 1939, however, owing to the increase in demand for "bottled gas" consisting predominantly of propane, the proportion of sales accredited to propane increased from 33 to approximately 36%, and a corresponding drop was recorded in propane–butane mixtures.

H. B. M.

BOOK REVIEW.

Sedimentary Petrography. By H. B. Milner. Pp. xxiii + 666. Thomas Murby & Co., London. 1940. Price 45*s*.

A new edition of this well-known work has been long overdue, and will be welcomed by all who knew, and had profited by, the second edition which was published in 1929. In his preface to this third edition, the author points out that unavoidable delay in producing it is offset by the opportunity afforded to review over a decade of international reactions to the subject, and to take care of the many activities, both in research and in its economic applications, which have marked its progress in the interim. It is in its application for the solution of many economic and industrial problems that Sedimentary Petrography may claim to be a comparatively new science, and it is very evident that remarkable advances have been made during the last ten years in the scope of such applications. This is well brought out by a comparison between the old and new editions, for whereas the former stressed more particularly the application of systematic petrography for the correlation of strata and subsurface geology, with an additional chapter on the study of soils, the latter embraces such subjects as: the asphalt industry (building construction), the technology of building, cement, ceramics, glass, highway construction, refractories, water supplies, and even criminology and industrial maladies.

Although the general arrangement of the subject-matter is not very different from that of the 1929 edition, many of the chapters have been expanded and embellished by new illustrations or diagrams, new chapters have been added, and in some cases the old phraseology altered and improved.

It would be impossible, within the scope of a brief review, to comment in detail on the many changes which have been effected, but for the sake of those already in possession of the old edition and who might wish to know whether the acquisition of the new volume was worth their while, some of the outstanding examples of new and valuable subject-matter may be referred to.

The chapter on " Sampling, Storage, and Records " has a new section on sampling from bulk for laboratory analysis, and the author goes to some length in stressing the necessity for guarding core samples from being interfered with by unauthorized persons.

Laboratory technique receives considerably more attention, and now occupies three chapters instead of one. There is a new section devoted to mounts of thin sections in media other than Canada balsam. The second edition made but a brief reference to the application of synthetic resins for this purpose, and the new volume deals with the subject in considerable detail, even to the extent of describing the method and apparatus required for preparing such resins in the laboratory. At first sight this might appear to be outside the legitimate scope of a book about petrography, and the more so because such resins are obtainable commercially ready made.

There are occasions, however, when the freshly made material is more convenient to use, especially for thorough impregnation, and for this reason a description of the method for preparing it is fully justified.

In the list of essential equipment for the treatment of incoherent rocks for the recovery of heavy minerals there is a change in the recommendations, in that sieves for special quantitative work are now referred to B.S.I. standards instead of those of the I.M.M.

Sieve analysis, which was only briefly referred to in the second edition, now receives considerable attention, about eight pages being devoted to the subject, and there is included a useful table giving the comparative apertures of the I.M.M., Tyler, A.S.T.M., and B.S. series of sieves, as well as some valuable hints on technique and the expression of results.

Elutriation is also dealt with more extensively than in the previous edition, a valuable contribution being the author's experience in the method of using Andrew's Kinetic Elutriator. There are also new paragraphs on air elutriation and centrifugal methods of separation.

A description of the Hallimond Electro-Magnetic Separator is another interesting addition. Although an excellent photograph of this apparatus is reproduced, the description would have been better illustrated by a line diagram, since the photograph fails to show some of the essential features. For instance, the position of the first and second separating magnets is hidden from view.

There is a new chapter which deals somewhat briefly with the application of *X*-rays, spectrum, fluorescence and micro-chemical methods of analysis. Within the scope of the nineteen pages devoted to these subjects, each of which might claim a volume to itself, nothing more could be expected than a bare outline of the special applications which such methods might have to sedimentary petrography. Actually the chapter falls short of expectations in this respect, since space has been used for generalized descriptions of apparatus and methods proper to these dissimilar sciences which might perhaps have been more usefully employed in specifying their special application to the true subject-matter of the book. Nevertheless, as the author himself points out in a concluding paragraph, those interested in the subject may obtain further details from the special publications which he cites in footnotes to the chapter.

The chapter on the " Microscopical Examination of Sediments " has been considerably extended and brought up to date. The old diagrams illustrating pleochroism, extinction, refractive index, and interference phenomena have been replaced by new and considerably improved illustrations. That the book is abreast of the time is evidenced by the use of " black-out " as a synonym for extinction, although the analogy would be somewhat strained if one spoke of parallel, oblique, or incline black-outs.

The chapter entitled " Quantitative Data " in the old edition is replaced by one called " Methods of Testing," which comprises a fair selection of standard methods taken from the publications of I.P.T., B.S.S., A.S.T.M., and other authoritative sources.

A characteristic and valuable feature of the old edition was the chapter on " Diagnostic Properties of Sedimentary Rock Minerals." This included the complete description of 72 minerals, 55 of which were illustrated, and brief descriptive notes on 25 others which were referred to as essentially sporadic and local minerals.

The new edition describes 131 minerals with illustrations of 61, the 25 sporadic examples being now included in the general category, with somewhat fuller descriptive details than heretofore.

The introductory pages of this chapter now contain illustrations of typical forms in the various crystallographic systems, and a complete list of all the minerals dealt with arranged according to their crystallization systems.

The chapters on " The Petrography of Consolidated Sediments," " The Principles of Correlation," and " Palæogeographical Problems " are not greatly different from the corresponding chapters in the older edition, although the tables of tendencies as regards occurrence, stability, and frequency, and the geochemical notes on the latter subject, have been considerably enlarged.

The second edition contained an appendix consisting of a set of tables in which some of the commoner detrital minerals were classified according to colour, refractive index, and behaviour with polarized light. This has been omitted in the new edition, and in its place are substituted a series of eight appendices, each representing a separate table listing essential properties such as system of crystallization, specific gravity, hardness, refractive index, etc., arranged in order of increasing numerical values.

This is a noteworthy improvement on the older arrangement as an aid to mineral diagnosis, and should prove of great value to students, both for ready reference and as a means of impressing the characteristic properties of the minerals on their memories.

The volume is completed by an extensive bibliography, both for the general subject and separately for some of the individual chapters, it is well and adequately indexed, and both the printing and binding are excellent.

In conclusion, it should be evident from the above that owners of the old edition of this work would be well advised to obtain possession of the latest edition if they wish to be up to date in their knowledge of the subject, whilst to those who approach the subject for the first time the book can be heartily recommended as one of the best at present available.

J. McConnell Sanders.

INSTITUTE NOTES.

February, 1941.

FORTHCOMING MEETINGS.

Tuesday, 11th March, 1941, at 5 p.m. at the Royal Society of Arts, John Adam Street, London, W.C.2. **“Octane Rating Relationships of Aliphatic, Alicyclic and Mononuclear Aromatic Hydrocarbons, Alcohols, Ethers and Ketones,”** by Dr. G. Egloff and P. M. van Arsdell. The Paper will be read by Dr. A. E. Dunstan.

Friday, 25th April, 1941. Annual General Meeting. Address by the President: **“Petroleum as a Raw Material.”**

ELECTION OF OFFICERS.

The Council has elected Professor A. W. Nash, M.Sc., M.I.Mech.E., to continue in office as President of the Institute for the Session 1941–42.

The following have been elected Vice-Presidents of the Institute for the Session 1941–42:

Ashley Carter, A.M.I.Mech.E.
C. Dalley, M.I.E.E.
F. H. Garner, Ph.D., M.Sc., F.I.C.
J. McConnell Sanders, F.I.C.
F. B. Thole, D.Sc., F.I.C.

ELECTION FOR COUNCIL.

In accordance with the Articles of the Institute, the following members of Council retire at the next Annual General Meeting in April, but offer themselves for re-election: A. F. Dabell, V. C. Illing, E. R. Redgrove, and W. J. Wilson.

One new nomination has been received on behalf of Robert Crichton, J.P. (General Manager, Scottish Oils Ltd.).

The Council has decided that, in view of present circumstances, a postal ballot will not be held. The names of the above five candidates will be submitted to the Annual General Meeting of members to fill five vacancies on the Council.

NEW MEMBERS.

The following elections have been made by the Council in accordance with the By-Laws, Sect. IV, para. 7.

Elections are subject to confirmation in accordance with the By-Laws, Sect. IV, paras. 9 and 10.

As Fellows.

Baars, Gerrit	England.
Smith, Reginald John	,,
Williams, Charles G.	,,

Transferred to Fellow.

ROBINSON, James T. T.	Trinidad.

As Members.

BUSH, Sydney Steve	England.
FOSSETT, Hubert	,,
JOHNSTON, Douglas Gordon	,,
MARGOSCHES, Charles G.	,,
WARD, Stanley Allen	,,

Transferred to Member.

HOLE, Herbert Wray	Canada.

As Associate Members.

BLANC-SMITH, William Leonard le	England.
BOOKLESS, Douglas Ernest	,,
DAVIDSON, Anthony Dryden	,,
GUNBY, Frank	,,
HUBLEY, John Stuart	Trinidad.
JEFFREYS, Lynn J. L.	S. Wales.
LANGDON, Charles Ernest	England.
O'CONNOR, D. D. Taaffe	Trinidad.
SMITH, Bernard William	England.

Transferred to Associate Member.

BANGERT, Norman Roy	England.
HOWARD, Frederick George	,,
KEACH, Raymond Harry	,,

As Students.

FARMAIAN, F. M.
FELLOWES, M. L.
GREEN, S. J.
HARTLEY, D. A.
JONES, R. E.
KEEN, S.
MORGAN, C. R.
OVER, J. R.
PEARCE, A. W.
SASSON, A.
SHORTHOSE, W. J.
SPENCER, F.
THURSTON, P.
WEAVER, A. G. T.
WHITEHEAD, R. C.
TWINN, G. B. ... ... England.

PAPERS IN THE *JOURNAL.*

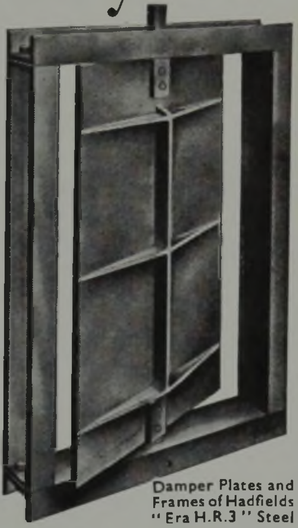
The following Papers are scheduled for inclusion in the March issue of the *Journal* :

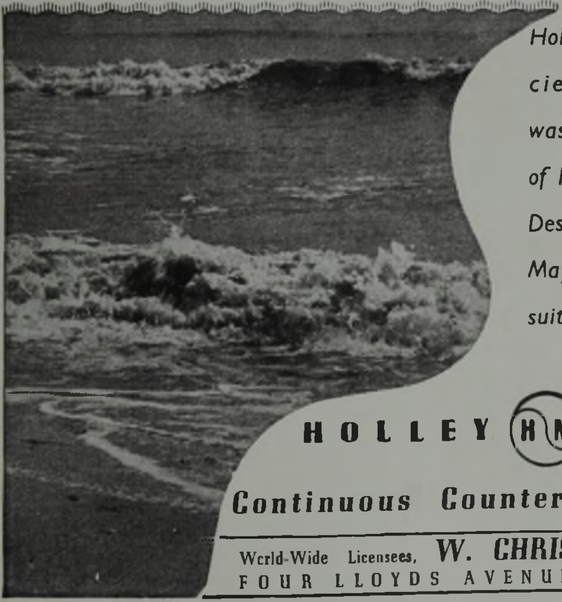
" The Use of Mineral Oils as Mosquito Larvicides," by H. D. Lord, Ph.D., F.I.C. (with contributed Discussion).

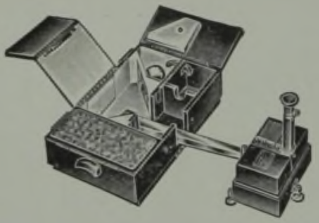
" The Investigation of High-Boiling Petroleum Oils by Adsorption Analysis," by B. C. Allibone.

" The Ollett Tube Cleaner." A note by E. A. Evans, F.C.S.

ARTHUR W. EASTLAKE,
Honorary Secretary.







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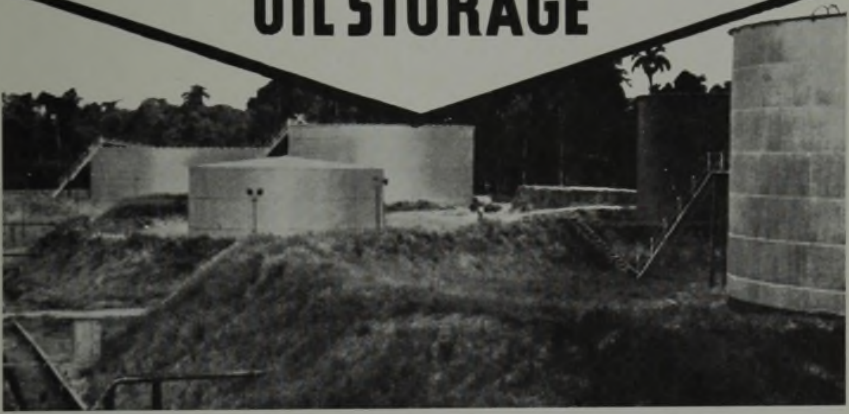

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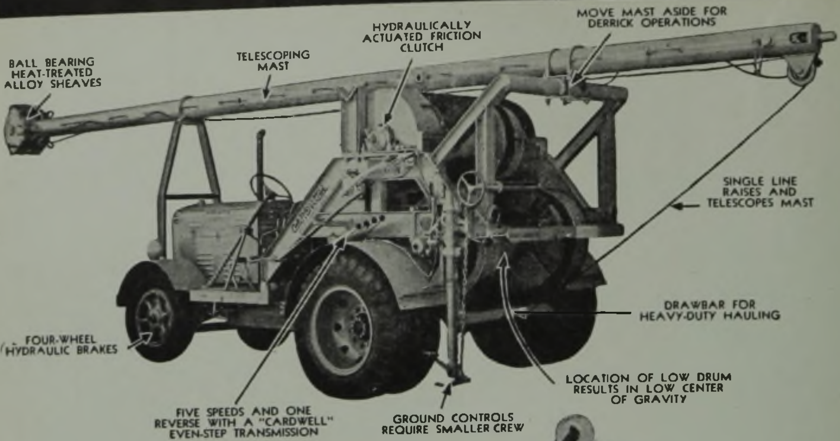

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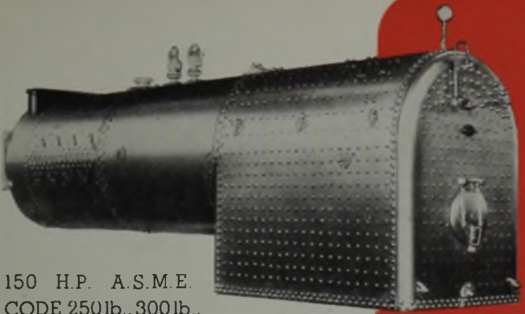

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