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EXTREME PRESSURE LUBRICANT TESTS WITH PRETREATED TEST-PIECES.*

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Summary.

Experiments with the Timken and Four-Ball lubricant testing machines are described, in which considerable increases in the breakdown loads of doped and undoped oils were obtained by pretreatment of the test-pieces with chlorinated or sulphurized dope or with hydrochloric acid. Running-in tests showed that the layer so formed was not removed by running under loads below the breakdown load. The pretreatment of the Cornell (or Falex) test-pieces had no apparent effect, but when these were hardened to a value comparable with that of the other machines, the breakdown loads became too high for the machine to measure. The formation of the layers is discussed briefly.

Introduction.

In the course of work on the production and testing of extreme pressure lubricants, experiments were carried out on three well-known lubricant-testing machines to investigate the formation of the film on bearing surfaces which is believed to underlie the phenomena of E.P. lubrication. The results, as is common with machines of this type, are not entirely self-consistent, but are considered to be of sufficient interest to justify publication.

The machines used were the Timken, the Four-Ball, and the Cornell (or Falex).

Timken Machine.

The work was commenced with the Timken machine.[1]

The test-pieces consist of a flat-rimmed cylindrical cup approximately 2 inches in diameter rotating at 800 r.p.m. in contact with a flat steel block. The block is pressed against the cup by means of a lever system which multiplies the thrust ten-fold, the results usually being given in terms of the weight applied to the load lever. The standard test procedure consists of starting the motor and applying the load at the rate of approximately 2 lb. per second. The machine is then allowed to run for 10 minutes under a constant load, a gallon of the lubricant being circulated over the test-pieces throughout the run. The test is repeated at various loads until the maximum load which gives a smooth scar on the test-block is found.

Types of Scar Obtained with Timken Machine.

Under the above conditions the results obtained at low loads are a smooth scar (Fig. 1) or a breakdown (Fig. 2). At high loads (50–100 lb.), breakdown, if it occurs, is more severe (Fig. 3), and other types of scars may also be obtained; these are narrow and rough (Fig. 4), wide and scored (Fig. 5), or wide and smooth (Fig. 6). Inspection of the test-cups

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† Imperial Chemical Industries, Ltd.

corresponding to Figs. 5 and 6 would reveal traces of metal " pick-up " indicating that a breakdown must have occurred during the run, although this was not indicated by a slowing down of the machine. It is clear, however, that the breakdown has been followed by recovery, since the damage to the surfaces has been repaired, the signs of breakdown being more obvious in Fig. 5 than in Fig. 6. When a severe breakdown, such as that shown in Fig. 3, takes place, the machine is slowed down considerably and the motor has to be stopped to avoid damage. The factors controlling the severity of breakdown cannot be said to be fully understood.

Rough scars, such as that shown in Fig. 4, were always accompanied by an appreciable darkening of the cup, which also became marked with circular lines parallel to its edge. Although completely satisfactory lubrication was not maintained, a high load was carried without the extensive damage to the test-pieces which accompanies a breakdown (Fig. 3).

The occurrence of these scars depended to some extent on the method of loading. Rapid application (*i.e.*, in less than 3 seconds) of a load of 100 lb. could produce a breakdown (Fig. 3) with most lubricants. Slower application might produce breakdown, breakdown followed by recovery (Figs. 5 and 6) or a narrow rough scar (Fig. 4). Very slow application (*i.e.*, in a time greater than 60 seconds) usually produced a narrow rough scar.

The changes in weight of the test-pieces accompanying the production of these types of scar are given in Table I. The oil used consisted of a mineral oil with a viscosity of 60 Redwood No. 1 seconds at 200° F. containing 2 per cent. of chlorine in the form of a chlorine-containing dope.

TABLE I.

Timken Machine—10-minute Tests on Doped Oil.

Load, lb.	Type of Scar.	Change in Weight of	
		Cup, mgm.	Block, mgm.
40	Smooth (Fig. 1)	− 8	− 1
100	Breakdown (Fig. 3)	+85	−232
100	Wide scored (Fig. 5)	+34	−109
100	,, ,, (Fig. 6)	− 8	− 2
100	Rough (Fig. 4)	−88	+ 4

Experiments with Previously Used Cups.

It was clear that the production of the narrow, rough scars was connected with the above-mentioned changes in the surface of the cup, and it appeared that these at least began to occur during the application of the load. Tests were therefore carried out by applying the load before starting the motor; this had the expected effect of causing a marked reduction in the load carried by the doped and undoped oils, and also eliminated both the wide scars showing signs of recovery (Figs. 5 and 6) and the narrow, rough scars.

When rough scars were obtained in 10-minute tests under a load of

100 lb. with the standard method of loading, the cup suffered an appreciable loss in weight, as shown in Table I. This, however, did not correspond to a change in diameter of more than 0·01 mm. (*i.e.*, 0·02 per cent.) and it was therefore possible to use the cups again for further tests. This was done with both doped and undoped oils, and the results are given below in Table II.

TABLE II.

Timken Machine—10-minute Tests on Cups Previously Used at a Load of 100 lb. in Doped Oil.

Oil.	Load Applied.	State of Cup.	Maximum Load Withstood without Breakdown, lb.	Appearance of Scar.
Undoped.	Standard method.	Unused.	11	Smooth.
		Used.	18	,,
		Used and washed in water.	19	,,
Undoped.	Before starting.	Unused.	6	Smooth.
		Used.	21	,,
		Used and washed in water.	20	,,
Doped.	Standard method.	Unused.	100	Rough.
		Used.	100	,,
		Used and washed in water.	100	,,
Doped.	Before starting.	Unused.	20	Smooth.
		Used.	100	Rough.
		Used and washed in water.	100	,,

It should be mentioned that some of the scars obtained under higher loads with these cups were somewhat severely scored. The scoring in this case was clearly due to the fact that the surface of the cup was lined after having been used to produce a rough scar. However, the scar was still quite narrow.

It is seen that in spite of the irregularity of the surfaces, the loads withstood by the used cups were in some cases markedly higher than that withstood with new cups. This change was believed to be connected with the darkening of the cup, which was most likely to be due to reaction between it and the dope contained in the oil.

Experiments with Cups Heated in Pure E.P. Lubricant.

Some cups were therefore heated in the dope itself (chlorinated type). Preliminary experiments showed that a uniform layer could be obtained on them by heating for 5 minutes at 200° C. In order to remove any dope retained after treatment, the hot cups were cooled in mineral oil and were then washed in trichlorethylene. It was found that the layer then left on the cup was readily deliquescent (Fig. 7*b*); some of the cups were therefore further washed in water and dried. The cups were all left under oil or covered with grease in the period between treatment and use.

The results obtained with cups treated in this way are given in Table III. It is seen that the results obtained with those cups heated in the dope

TABLE III.

Timken Machine—10-minute Tests on Cups Previously Treated for 5 Minutes at 200° C. in Pure E.P. Lubricant.

Oil.	Load Applied.	State of Cup.	Maximum Load Withstood Without Breakdown, lb.	Appearance of Scar.
Undoped.	Standard method.	Untreated.	11	Smooth.
		Treated.	80	"
		Treated and washed in water.	19	"
Undoped.	Before starting.	Untreated.	6	Smooth.
		Treated.	79	Slightly scored.
		Treated and washed in water.	20	Smooth.
Doped.	Standard method.	Untreated.	100	Rough.
		Treated.	100	"
		Treated and washed in water.	100	"
Doped.	Before starting.	Untreated.	20	Smooth.
		Treated.	100	Rough.
		Treated and washed in water.	58	Smooth.

were again higher than those obtained with the untreated cups, the figures obtained with those which had not been washed in water being particularly high. The scars obtained when breakdown did not occur were narrow

TABLE IV.

Timken Machine—Tests of Two Types to Determine the Maximum Load Withstood after Running-in for 1 Hour.

(*a*) One 10-minute run with the run-in cup on a new block-face.
(*b*) A series of 2-minute runs with the same run-in cup and a new block-face each time, the load being increased in steps of 2 lb.

Oil.	State of Cup.	Load during Running-in, Applied by Standard Method, lb.	Tests after Running-in.			
			Type.	Load Applied.	Maximum Load Withstood without Breakdown, lb.	Appearance of Scar.
Undoped.	Untreated.	5	*a*	Standard method.	10	Smooth.
		5	*a*	Before starting.	10	"
	Treated.	15	*a*	Standard method.	46	Smooth.
		15	*a*	Before starting.	50	"
		15	*b*	" "	42	"
	Treated and washed in water.	15	*a*	Standard method.	50	Smooth.
		15	*a*	Before starting.	35	"
		15	*b*	" "	26	"
Doped.	Untreated.	33	*a*	Before starting.	60	Smooth.
	Treated.	50	*a*	" "	100	Rough.
	Treated and washed in water.	50	*a*	" "	100	"

and quite smooth (Fig. 8) except at the higher loads, when roughening took place to an extent which was rather variable.

Some tests lasting for longer periods were also carried out with cups treated in this way, and the results are given in Table IV. These were all run-in for an hour, and were then used for two types of test. The first was to determine the breakdown load of the oil with a run-in cup, and the second to find the result of running-in with gradually increasing loads.

Increases in the load withstood by undoped oil with treated cups are again shown.

Experiments with Cups Treated with Hydrochloric Acid.

One of the most active products of the high-temperature decomposition of chlorinated dopes is hydrochloric acid, and some tests were therefore carried out with cups which had been treated with it. The cups were immersed in acid of ten normal strength at 50° C. for 5 minutes, followed by washing in water. The results are given in Table V, and will be seen to show the same tendency as those obtained with cups which had been treated with the dope.

TABLE V.

Timken Machine—Tests on Cups Treated for 5 Minutes at 50° C. in 10N·HCl *and Washed in Water.*

After running-in, only one 10-minute run was carried out on each cup.

Oil.	Conditions of Running-in (Standard Loading).	Load Applied.	Tests after Running-in.	
			Maximum Load Withstood without Breakdown, lb.	Appearance of Scar.
Undoped.	None.	Standard method.	19	Smooth.
	,,	Before starting.	20	,,
	1 hr. at 15 lb.	,, ,,	40	,,
Doped.	None.	Standard method.	100	Rough.
	,,	Before starting.	78	Partly rough.
	1 hr. at 15 lb.	,, ,,	100	Rough.

FOUR-BALL MACHINE.

In view of the well-known lack of agreement between the results given by different lubricant testing machines, similar experiments were also carried out with the Four-Ball machine. The test-pieces of this consist of ½-inch steel balls of ball-bearing quality; one of these is rotated at 1500 r.p.m. in contact with the other three, which are clamped together in a cup containing the lubricant under test. The load is applied before the commencement of the test, which is of 1 minute's duration.

Full descriptions of this machine are available elsewhere.[2, 3, 4] The breakdown load of a lubricant is found in terms of the load required to produce seizure 2½ seconds after the test is started,[5] and the maximum load withstood for a minute can also be obtained.[3, 4] If the diameters of the scars on the three stationary balls at the end of each test are plotted against the load, it is also possible, with some oils, to determine the pressure at which lubrication is re-established after seizure. From Fig. 12 it will be seen that if double logarithmic paper is used, the lines representing constant pressure are straight. The graphs given therein

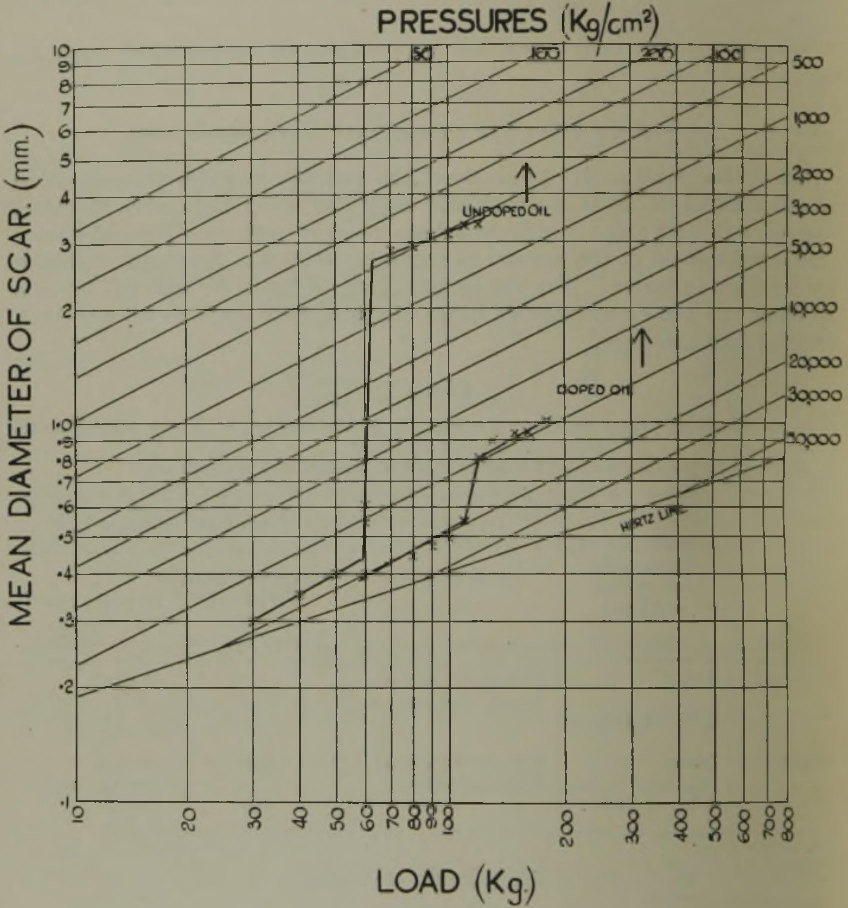
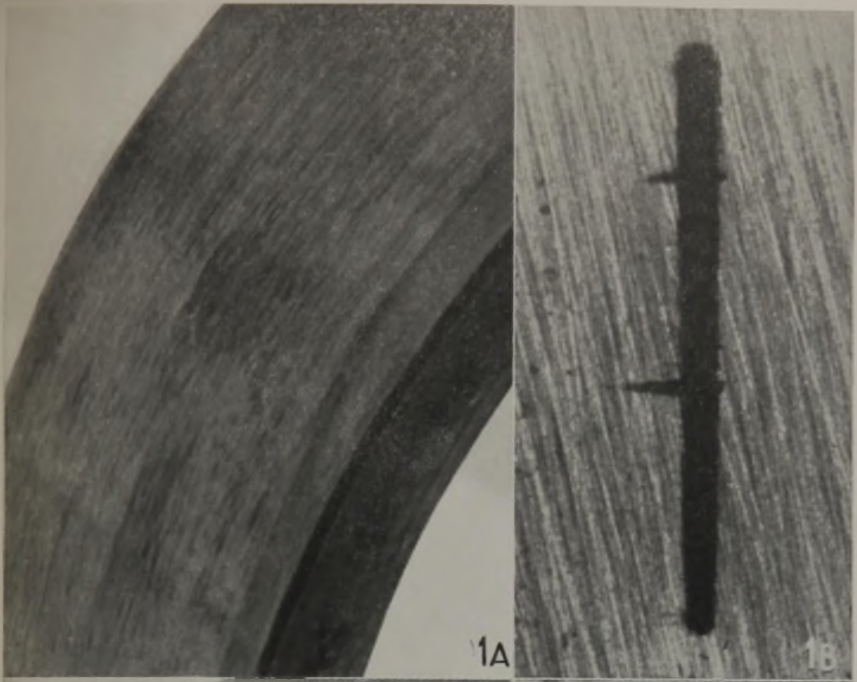


Fig. 9.

DIAMETER OF SCAR ON THE LOWER BALLS AFTER A TEST PLOTTED AGAINST THE LOAD. FOUR BALL MACHINE.

show that with both doped and undoped oils the diameter of the scar at loads too low to produce seizure is nearly the Hertz value for the elastic deformation of the balls. After seizure, which is marked by a sudden increase in the diameter of the scar, the graph is often parallel with the lines of constant pressure, indicating that over a range of loads the pressure at which lubrication is re-established after seizure is constant. At still



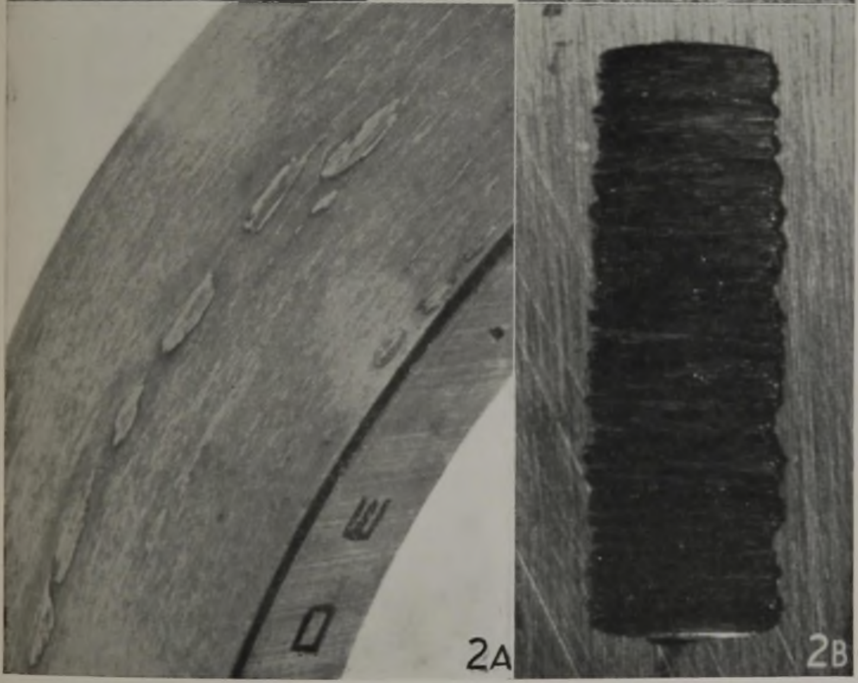


Fig. 1 (*a*) and (*b*).

SMOOTH SCAR OBTAINED WHEN NO BREAKDOWN OCCURS AT LOW LOADS WITH A MINERAL OIL.

Fig. 2 (*a*) and (*b*).

[illegible] INERAL OIL.

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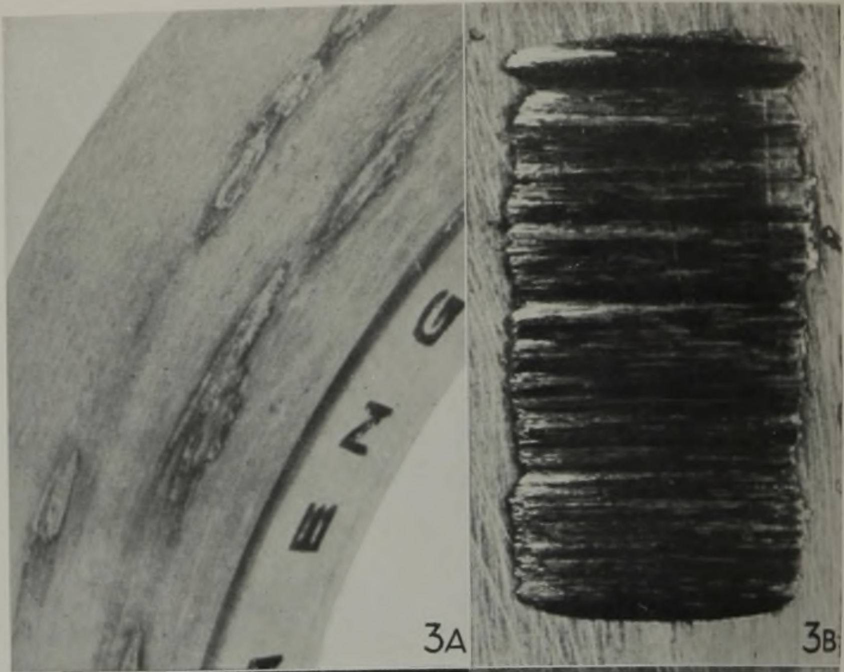


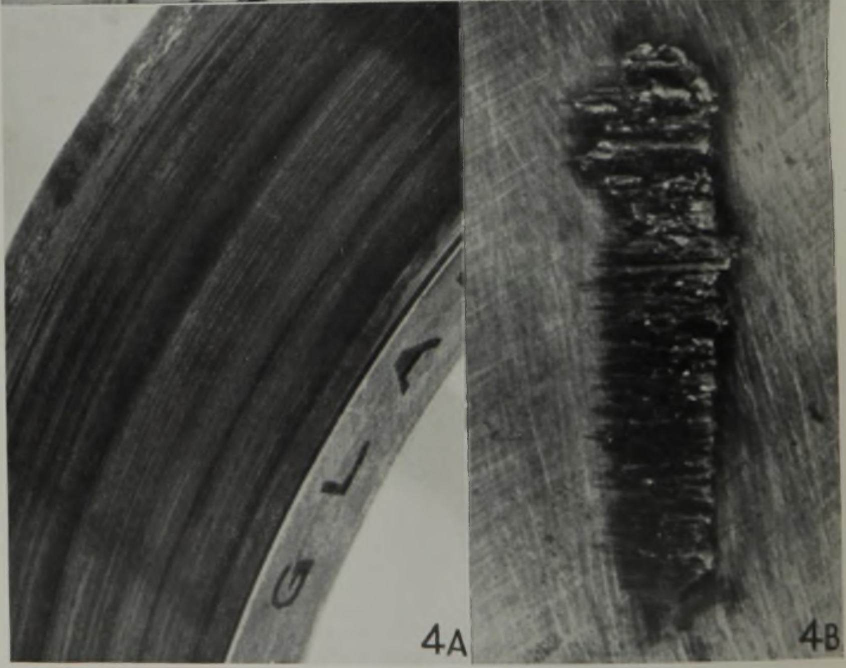
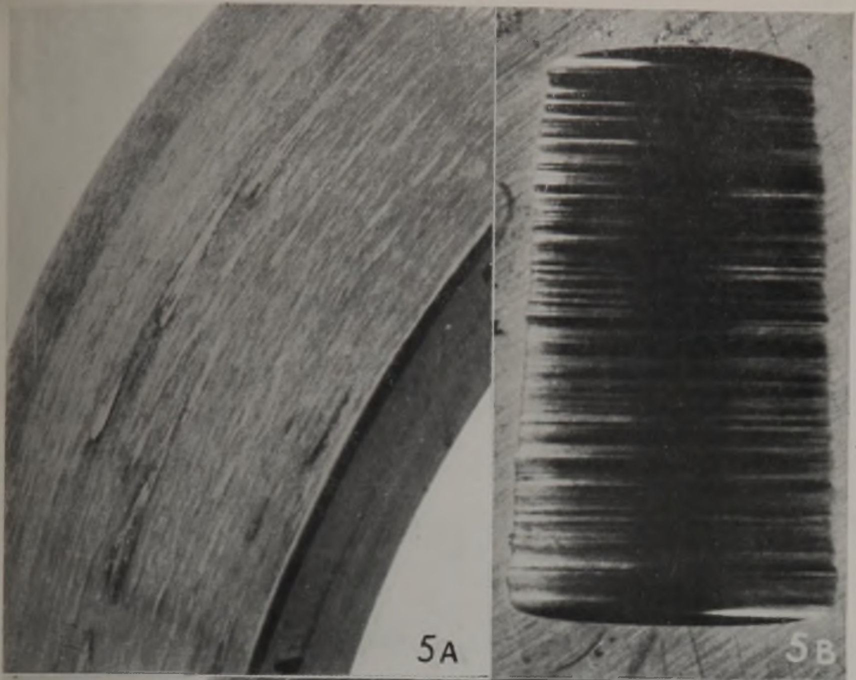


FIG. 3 (*a*) AND (*b*).

BREAKDOWN WITH DOPED OIL UNDER HIGH LOAD.

FIG. 4 (*a*) AND (*b*).

ROUGH SCAR OBTAINED WITH DOPED OIL UNDER 100 LB. LOAD.



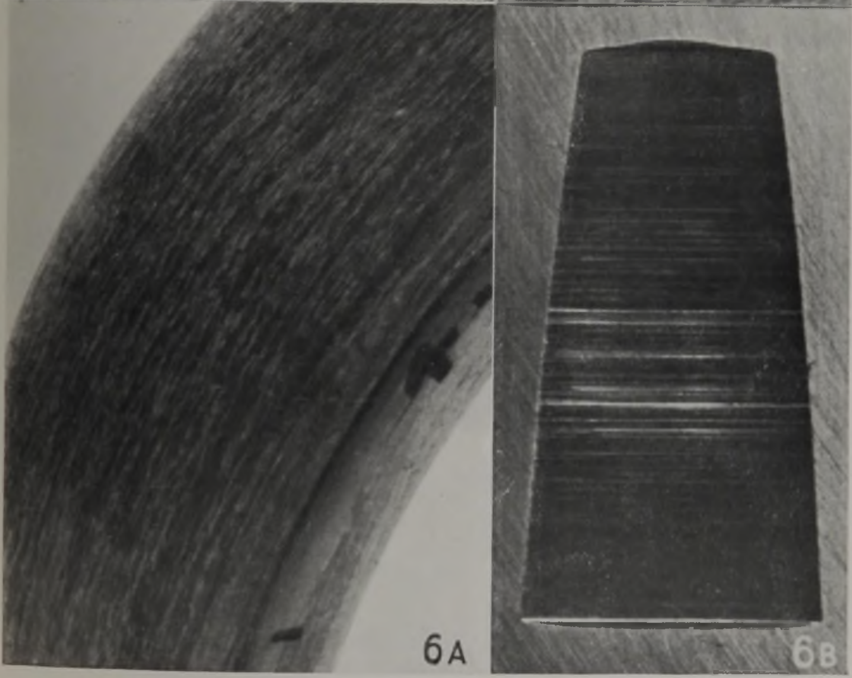
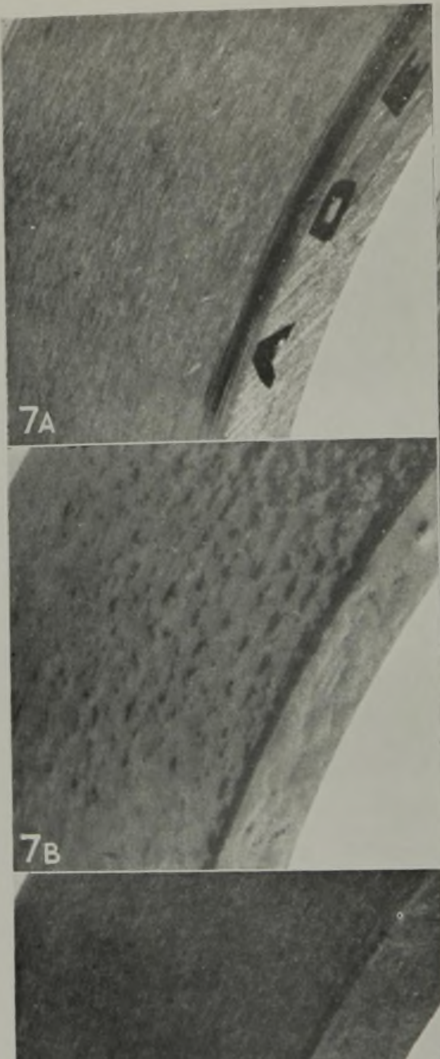


Fig. 5 (*a*) and (*b*).
WIDE SCORED SCAR OBTAINED WITH DOPED OIL.

Fig. 6 (*a*) and (*b*).
WIDE SMOOTH SCAR OBTAINED WITH DOPED OIL.



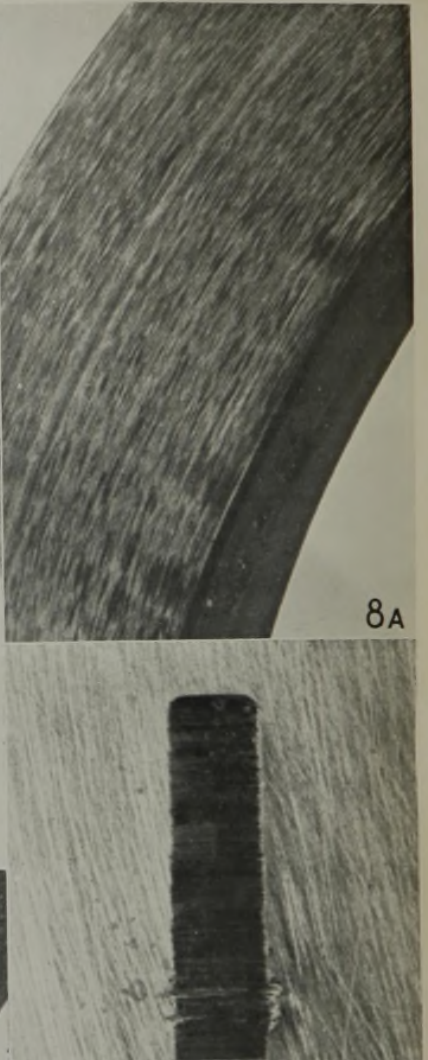


Fig. 7.

(*a*) UNUSED CUP.

(*b*) CUP HEATED FOR 5 MINUTES AT 200° C. IN CHLORINATED DOPE AND WASHED IN OIL AND TRICHLORETHYLENE, SHOWING DELIQUESCENT LAYER.

(*c*) CUP TREATED AS IN (*b*) AND THEN WASHED IN WATER AND DRIED.

Fig. 8 (*a*) and (*b*).

SMOOTH SCAR OBTAINED WITH UNDOPED OIL UNDER A LOAD OF 70 LB. APPLIED IN THE STANDARD WAY USI[illegible] IN WATER.

higher loads the welding together of the balls may occur. The re-establishment of lubrication does not always take place at a pressure which is completely independent of the load, but it usually varies sufficiently slowly for the mean value to be used as an indication of the intensity of seizure.

Tests with the Rotating Ball Treated before Use.

Tests were carried out with the rotating ball treated in various ways, and the results are given in Table VI.

TABLE VI.

Four-Ball Machine.

Dope Used (expressed as % Cl or % S in doped oil).	State of Top Ball.	2½-Second Seizure Load, kg.	Pressure after Seizure, kg./cm.^{2}
None.	Untreated.	75	500
	Treated in chlorinated dope.	154	500
	Treated in chlorinated dope and washed in water.	137	500
	Treated in HCl.	159	500
	Treated in sulphurized dope.	122	500
3% chlorine.	Untreated.	135	10,000
	Treated in chlorinated dope.	168	13,000
	Treated in chlorinated dope and washed in water.	200	12,000
	Treated in HCl.	258	15,000
	Treated in sulphurized dope.	186	12,000
1% sulphur.	Untreated.	160	3,000
	Treated in chlorinated dope and washed in water.	174	10,000
	Treated in HCl.	190	8,000
	Treated in sulphurized dope.	220	10,000

The oils used with this machine were a mineral oil of viscosity 140 Redwood No. 1 seconds at 200° F., and the same oil containing 3 per cent. of chlorine or 1 per cent. of sulphur in the form of a chlorinated dope and a sulphurized fatty oil, respectively. The reason for using a lower percentage of chlorinated dope on the Timken Machine was merely that the maximum load of that machine was attained rather readily with higher proportions of this material.

The treatment of the balls with both the chlorinated and the sulphurized dopes consisted of heating for 5 minutes at 200° C., followed by cooling the balls in mineral oil and washing them in trichlorethylene. Some of the balls treated with the chlorinated dope were then washed in water and dried; and others were used without further treatment. Washing of the sulphurized balls with benzene, petroleum ether, or water made no difference to the results obtained with undoped oil, and so was not used for the tests with doped oil. The treatment with hydrochloric acid consisted of placing the balls in 10N-acid for 5 minutes at 50° C., followed by washing them in water.

It is seen that all the kinds of pretreatment gave rise to marked increases in the 2½-second seizure loads of both doped and undoped oils. Tests with all four balls treated in the same dope gave the same results as were obtained when only the rotating ball had been treated.

Cornell (or Falex) Machine.

The test-pieces used in this machine consist of a piece of ¼-inch steel rod of hardness 5C Rockwell rotating between two V-grooved test-blocks of hardness 10–15C Rockwell. The test-pin is fitted into a chuck which can be rotated at 290 r.p.m., and a piece of thick brass wire placed through holes in the chuck and pin serves to transmit the driving-torque to the pin. The blocks are held against the pin in a pair of jaws by a spring-loading system which is operated by rotation of a ratchet wheel. A device is fitted for rotating the ratchet wheel automatically, and this gives a steadily increasing load, the rate of increase being approximately 1000 lb. per minute. Abrasion of the pin tends to reduce the load on the test-bearing for a given setting of the ratchet wheel, and consequently a constant rate of rotation of the ratchet wheel may not then give a constant rate of increase of load. A hydraulic system is provided to give continuous indication of the friction.

One usual type of test on this machine consists of starting the motor with a small load applied, which is allowed to increase automatically up to 500 lb. The automatic device is then detached to give a running-in period of 5 minutes at this load, after which the load is increased automatically again.

Breakdown of the lubricant is marked by a rapid rise in the frictional torque, and if very severe, by shearing of the brass wire or of the test-pin near the hole through which the wire is fitted.

Abrasion tests in which the load is maintained at a constant value may also be carried out, the abrasion being measured by observing the angle through which the ratchet wheel in the loading device has to be turned in order to maintain the load at a constant value. Tests may also be made in which the load is applied before the motor is started.

All three types of test were carried out with test-pins treated in the chlorinated dope as described for the other two machines, but no significant difference in results was obtained as compared with untreated pins. It was thought that this difference between the Cornell and the other two machines might be due to the relatively low hardness of the test-pieces, and a batch of them were therefore hardened to 60C Rockwell. With these pins the breakdown loads appeared to be beyond the range of the machine, whether the pins were treated or not, and the effects of treatment could therefore not be observed.

Discussion.

Timken Machine.

Treatment of the cups for the Timken machine by (1) running for 10 minutes under a load of 100 lb., whilst lubricated with oil containing the chlorinated dope, (2) heating in the dope, (3) heating in hydrochloric acid, considerably increased the load withstood when they were subsequently used with undoped oil, whether the load was applied before or after starting the motor. With doped oils an increase could not be observed when the standard method of loading was employed, as the maximum load was withstood by untreated cups; but similar effects were noticed when the motor was started with the load on.

A breakdown load of 20 ± 2 lb. was obtained with undoped oil in a number of ways. Cups treated by running for 10 minutes at 100 lb. in doped oil, or by heating in the dope and washing in water, or by heating in hydrochloric acid and washing in water, all gave this value with either method of loading; and it was also given when testing doped oil with untreated test-pieces by starting the motor with the load on. This agreement may be a coincidence, or it may be connected with the nature of the layers formed by the action of chlorine or hydrochloric acid on a Timken cup.

Four-Ball Machine.

The results obtained with the Four-Ball machine confirmed the increases in breakdown load obtained by treatment on the Timken machine. They also showed that treatment of the top ball does not alter the pressure at which lubrication was re-established by an undoped oil after seizure.

The Temperature Distribution in the Test-pieces.

All the machines described herein have a rotating test-piece in contact with one or more stationary ones. Under the conditions which prevail during the early parts of the tests, the surfaces are separated by a thin layer of oil. The load is carried on local high spots, and the temperatures at the tops of these may reach the melting point of the metal even under comparatively low loads.[6] The temperature of the metal immediately below them will depend on the amount of heat generated at their tops and the rate at which it is conducted away into the body of the test-piece.

The stationary test-pieces are continuously heated during a test, but the rotating ones are cooled by contact with the oil for an appreciable part of each revolution. The metal immediately below the high spots of the stationary test-piece may consequently be expected to be at a higher temperature than the corresponding portions of the rotating ones. It is therefore possible, when a sufficiently high load is used, for this portion of the stationary test-pieces to reach a temperature near the melting point, whilst the corresponding portions of the rotating test-piece are comparatively cool, and welding of metal on to the rotating test-piece can then occur. This occurs when an undoped oil breaks down on each of the three machines described.

When a doped oil is used, the evidence available shows that the coefficient of friction is nearly the same as for an undoped oil in the early part of the test; the presence of the dope cannot therefore affect the temperature distribution appreciably.

Further, loads can be withstood by doped oils which would produce welding with undoped ones. It thus appears that when a dope is used, the metal immediately below the high spots of the stationary test-pieces can be near its melting point without seizure taking place.

The occurrence of such high temperatures in the stationary test-piece without seizure may only lead to an extension of the polishing which occurs at low loads. If the conditions are appropriate, however, it appears that it may lead to appreciable flow of metal from the point of highest temperature in the direction of motion of the test-piece. This is believed to be what takes place when rough scars, such as that shown in Fig. 5, are formed.

Formation of Surface Layers.

The results show that high loads may be withstood by undoped oils without seizure as a result of the previous reaction between the dope and the metal, to form a layer on its surface. This suggests a mechanism for the effects observed with doped oils. These dopes do not react appreciably with metal at ordinary temperatures, and so the layer can only be formed by pretreating the test-pieces, or as a result of the heating they receive during the early part of a test using a doped oil. In the latter case the layer is unlikely to be fully formed before some damage has occurred.

The increased breakdown loads obtained with treated test-pieces may be due to the fact that the full protective layer has been formed in advance. It is therefore possible that improved results might be obtained in practice with doped oils by heating bearing surfaces in the dopes before use. The practice mentioned by Clayton [3, 4] of "running-in" gears with powerful E.P. lubricants and then using them with a milder one is an approximation to this; but even in this case the layer produced by the powerful dope cannot be fully formed without running the risk of damage due to local overheating, and pretreatment with it might give improved results.

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SOME REMARKS REGARDING THE TESTING OF ENGINE LUBRICANTS.*

By C. A. Bouman.†

Synopsis.

Only a few of the characteristics of motor oils can be determined by physical and chemical analysis.

Oxidation tests do not represent the various processes of deterioration taking place in the engine; such great differences exist between processes taking place in various parts of the engine that oils are not rated in the same order of merit for them all. Also, deterioration of engine oil is often a matter of contamination with combustion products, particularly in C.I.-engines.

Carbon tests are only of some value with regard to carbon formation in the combustion space; not, however, with regard to carbon formation in piston-ring grooves or to the tendency to stick rings.

Engine tests are necessary, but wearisome, as the degree of reproducibility required is often too small to allow of reaching a sound conclusion from only a few tests. In particular, the test conditions must also be chosen so as to give a good correlation with those conditions in practice for which an oil is intended.

The reliable judging of lubricating oils is still a source of much worry to the users of oils, to the manufacturers of engines, and to the manufacturers of oils. The most characteristic property of a lubricating oil—viz. its viscosity at various temperatures—can be determined accurately in a very simple way, but there remains the extensive and difficult field of investigation of those properties of an oil which make it either capable or incapable of withstanding the high temperature conditions at various points in the engine. The following remarks mainly concern this field of investigation. Other important properties of engine oils, such as their behaviour at low temperatures, oil consumption, anti-wear characteristics, etc., are left out of account here.

A. Laboratory Oxidation Tests Compared with Practical Results.

For a long time the efforts of many investigators have been directed towards the development of laboratory oxidation tests for lubricating oils. With the aid of these tests it was intended to predict whether a given oil would be suitable for certain types of internal-combustion engines or not. As a result of these attempts, many laboratory oxidation tests, more or less standardized and more or less officially approved, are available or have been suggested. However, it cannot be said that great success has attended these efforts, the main reason being that the deterioration of the oil in the laboratory test and that occurring in internal-combustion engines are of a different nature.

The deterioration processes under high temperature conditions, to which the lubricating oil is exposed in the engine, are of a very complicated character. This applies particularly to those processes which finally result

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† "Delft" Laboratory, Royal Dutch Shell.

in the formation of carbon deposits in the cylinder and on the piston. Further, as is well known, these processes depend very much on engine conditions, especially engine temperatures. When the engine conditions are changed, the intensities of the various processes (oxidation, polymerization, carbonization) to which the lubricating oil is exposed in the engine will also change, and there will be transitions from one process to another. These changes in intensity may be different for various lubricating oils. Testing various oils in one engine under certain test conditions will establish a certain relative rating for the oils. Testing the same oils in another engine or under other engine conditions may establish quite another relative rating.

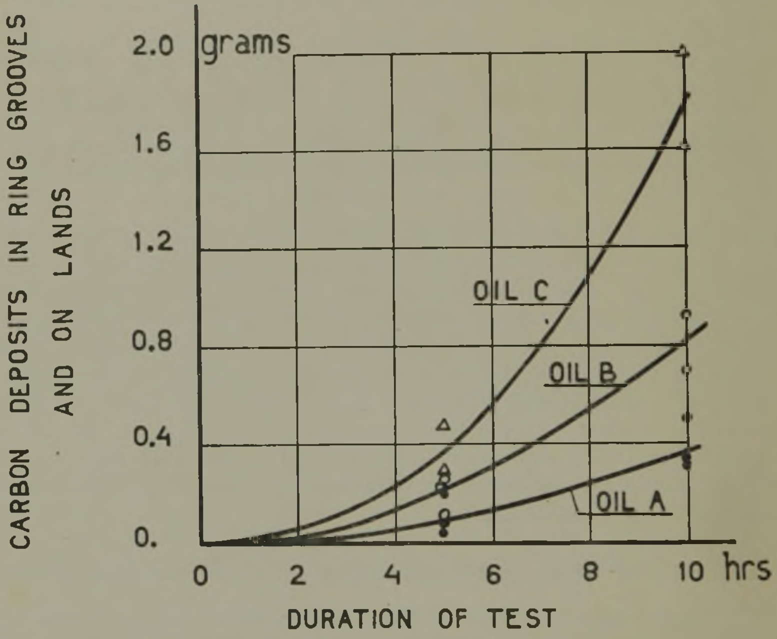

FIG. 1.

CARBON DEPOSITS IN RING GROOVES AND ON LANDS.

Further, it has been confirmed by many results, both in the engine laboratory and in practice, that when testing various oils in a certain engine under certain test conditions, a certain relative rating for the oils as regards the formation of deposits and other undesirable products will be established for one certain spot in the engine; yet in the same engine and under the same test conditions the same oils may show quite another rating for another spot in the engine. Thus, for instance, oils showing a certain relative rating as regards carbon formation in the ring grooves may show quite a different relative rating as regards carbon formation in the combustion space. An example is given in Figs. 1 and 2, showing the results of tests with a single-cylinder horizontal gasoline engine with ethylene glycol cooling, running on three different oils, *A*, *B*, and *C*.

At best the application of the results of laboratory oxidation tests should be restricted solely to a certain number of the processes taking place in the engine, whereby the temperatures prevailing during a test should be adjusted as closely as possible to the temperature existing or supposed to exist in that part of the engine where the reactions under consideration occur. However, for the study of the more complicated processes occurring in the engine (*e.g.*, ring-sticking) the investigator will necessarily come to a point where he recognizes the desirability of adjusting not only the temperature of his oxidation test to the desired value, but also the mechanical conditions of the test to those occurring in the engine. This leads to the adoption of so-called semi-technical apparatus.

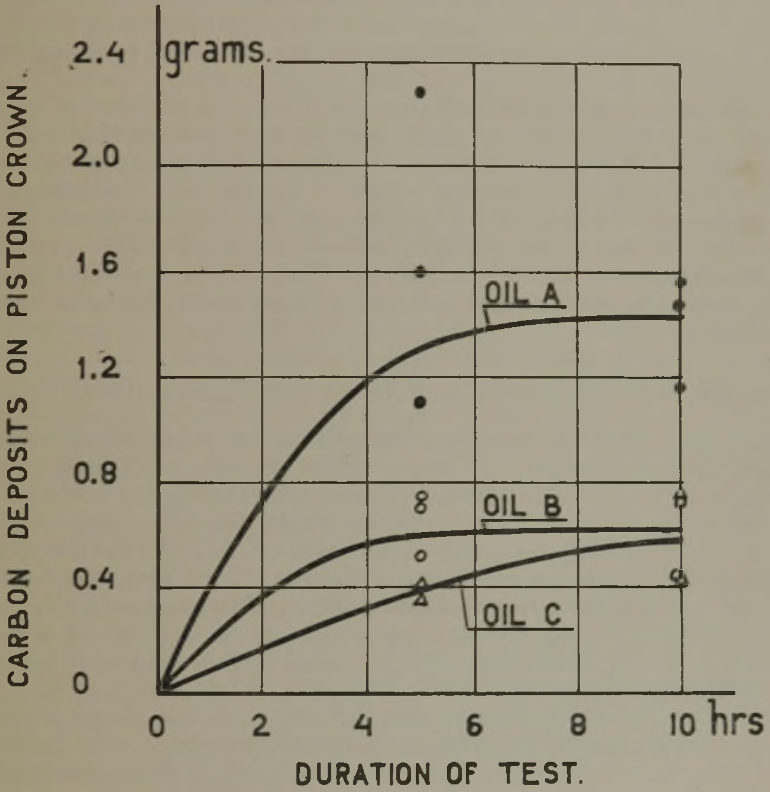

FIG. 2.

CARBON DEPOSITS ON PISTON CROWN.

In many cases the deterioration of the crankcase oil, as measured by the percentage of asphaltenes contained in it, may show a fair correlation with several laboratory tests at moderate temperatures. However, for many engines deterioration of the crankcase oil cannot be measured only by the percentage of asphaltenes contained therein, since in these engines (especially diesel engines) the contamination of the lubricating oil in the crankcase mainly consists of products from incomplete combustion (soot, carbon, lacquer, etc.) of the fuel in the combustion space, which products have been absorbed by the lubricating oil on the cylinder wall and pass along the piston into the oil in the crankcase. It is quite clear that since an oxidation test only gives data regarding the deterioration of the oil itself, it cannot give any data regarding the contamination of the oil by products formed in the combustion chamber. Therefore, only a very slight value can be attributed to laboratory tests in regard to the judgment of engine-lubricating oils. These tests do not give a correct picture of the manner in which the lubricating oil is contaminated in the engine. At most it must be regarded as a lopsided picture, in which a certain kind of deterioration within the oil is accentuated, therefore giving only an indication in respect of phenomena which, in practice, are found to be relatively unimportant.

It should also be pointed out that the increase in viscosity of the oil during an oxidation test in many cases shows no correlation with its oxidizability. Nor does it show a correlation with practical conditions.

In special cases the laboratory oxidation test can indeed supply useful data regarding the suitability of an oil—*e.g.*, when the crankcase oils of large diesel engines act not only as a lubricant, but also as a cooling medium for the pistons with circulation cooling. The oxidation test then mainly bears on the behaviour of the oil, not as a lubricant, but as a cooling medium.

B. Laboratory Carbonization Tests and Semi-technical Tests.

In addition to the laboratory oxidation tests, which are mainly characterized by the fact that oxygen or air is blown through the oil under certain conditions, other tests have been developed, not so much with the object of measuring the resistance of the oil against deterioration at elevated temperatures, as of measuring the quantity of residue left behind after the oil has been exposed to high temperatures. As examples, the various carbonization tests (Conradson, Ramsbottom) may be mentioned. In the combustion space of an engine the temperatures are so high that it is quite impossible for any lubricating oil to withstand these temperatures without carbonization. Accepting the fact that any oil will carbonize at these temperatures, it nevertheless remains desirable that the oil will leave as small a residue as possible after the carbonization process. To a certain degree the residues found for these tests are a criterion of the volatility of the oil, especially of its heavier components.

Although these carbonization tests may give an indication of the behaviour of the oil when exposed to the combustion temperatures in the combustion space, they cannot give reliable information as regards its tendency to form carbon deposits in other parts of the engine—*e.g.*, in the piston-ring grooves. Moreover, the practical value of the carbonization tests should not be over-estimated, because after a certain quantity of carbon deposit has formed in the combustion space, an equilibrium is obtained : the surface of the carbon deposits has then attained a temperature at which the newly formed carbon particles burn away.

As an intermediate test between oxidation tests and carbonization tests, we may consider the so-called Delft " baking " test, developed by J. E. van Hinte of our Laboratory. With this test about 0·4 gram of the oil to be investigated is submitted to various constant temperatures (270–290° C.) for a certain time (about 40 minutes) in a small open aluminium cup. At the end of the test the loss by evaporation and the residue (insoluble in non-aromatic gasoline 60/80) are measured.

Another test on the same lines is the Wright Aeronautical Corporation's oxidation cup test.[1] The Delft "baking" test is now being modified by placing small steel discs on the oil layer at the bottom of the cup, so that it is now a combined baking and sticking test. In a few cases satisfactory correlation was found between the results of the baking test and the results of ring-sticking tests in the engine.

Further development of semi-technical testing equipment would be directed towards realizing in the apparatus as closely as possible the physical and mechanical conditions prevailing in an engine. It will be found in the long run, however, that tests on actual gasoline or diesel engines are not only simpler, but also far more reliable than tests with such apparatus.

C. Engine Tests.

In most cases, especially when the tendency of the oil to cause ring-sticking is to be studied, testing in the engine is the only way to obtain reliable information regarding the quality of the oil. Unfortunately, the engine tests also present considerable difficulties, mainly for two reasons. In the first place, when preliminary testing of aviation oils and of marine diesel-engine oils is conducted in relatively small gasoline and diesel engines, the question arises whether the results obtained with such small engines are applicable to the actual engines in practice. Secondly, the reproducibility of engine-fouling tests is often unsatisfactory, which fact, of course, tends to make the results confusing.

As to the first point, it has been found that when marine diesel-engine oils are tested in small diesel engines, and when aviation oils are tested in small gasoline engines, the classification of various lubricating oils as determined in the small oil-testing engines generally agrees to a satisfactory extent with the classification of the same oils in practice.[2]

In order to shorten the duration of the tests, the engine conditions may be made somewhat more severe than those occurring in practice. It has been found that when these changes are kept within reasonable limits, the relative classification of the oils is not affected too much.

The most difficult and at the same time most important point in the engine-testing of lubricating oils is the degree of *reproducibility* which can be obtained when tests are repeated under conditions as nearly identical as possible. A few remarks concerning this problem may be given with regard to determining the tendency to ring-sticking and carbon formation on the piston. First of all, it is important to note that an agglomeration of fouling products from the oil (together with contamination products from combustion) formed during a relatively long running time constitutes the tangible result of a test. In this respect there is a marked difference between the testing of lubricating oils and, for instance, the testing of the ignition quality of diesel fuels. In the latter case the result of the test is, in principle, obtained during only one cycle of the process, in the course of which the product (fuel) to be investigated is consumed. A new charge is supplied for the next cycle. Thus, no disturbing influences from previous cycles on the results are encountered. On the contrary, the accuracy can be improved by taking the average results of a great many cycles.

When combustion phenomena are studied, such as the tendency of fuels containing residual components to form carbon deposits on some spot of the combustion space, the results are an agglomeration of products formed over a long period, and it is known that the reproducibility of tests of that kind

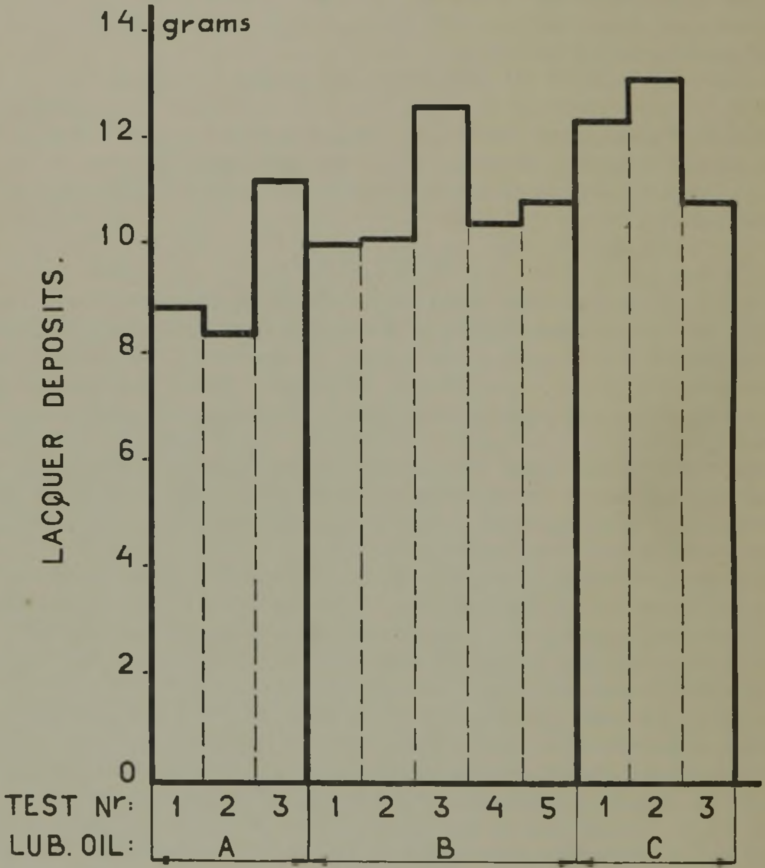

Fig. 3.

FAIR REPRODUCIBILITY OF LACQUER TESTS WITH AIR-INJECTION DIESEL ENGINE AT LOW LOAD.

is smaller than that of ignition quality measurements. Such unfavourable circumstances are always met with during tests with engine-lubricating oils. As regards ring-sticking tests, there is a further reason for the lack of reproducibility—viz., the fact that the ring-sticking phenomenon depends to a very great extent on the mechanical conditions of the piston and piston-rings. As such may be mentioned the side clearances of the piston-rings in their grooves, the elasticity (radial pressure) of the rings, the initiation of ring-sticking by adventitious accumulation of ring-blocking material somewhere along the ring circumference, the circumstance whether ring-sticking starts at the gap or at another point of the ring circumference, the degree to which heat-flow and blow-by are affected by initial ring-sticking, the thermal distortion of piston and cylinder, oil consumption, etc. All these factors cannot be kept under absolute control during a test. Also difficulties may arise as to the interpretation of the results, when, for instance, only the gap of the ring is stuck (both ends), whilst the other parts of the circumference are still free, and further, when a ring is still "just free" or is only "slightly sticking," etc.

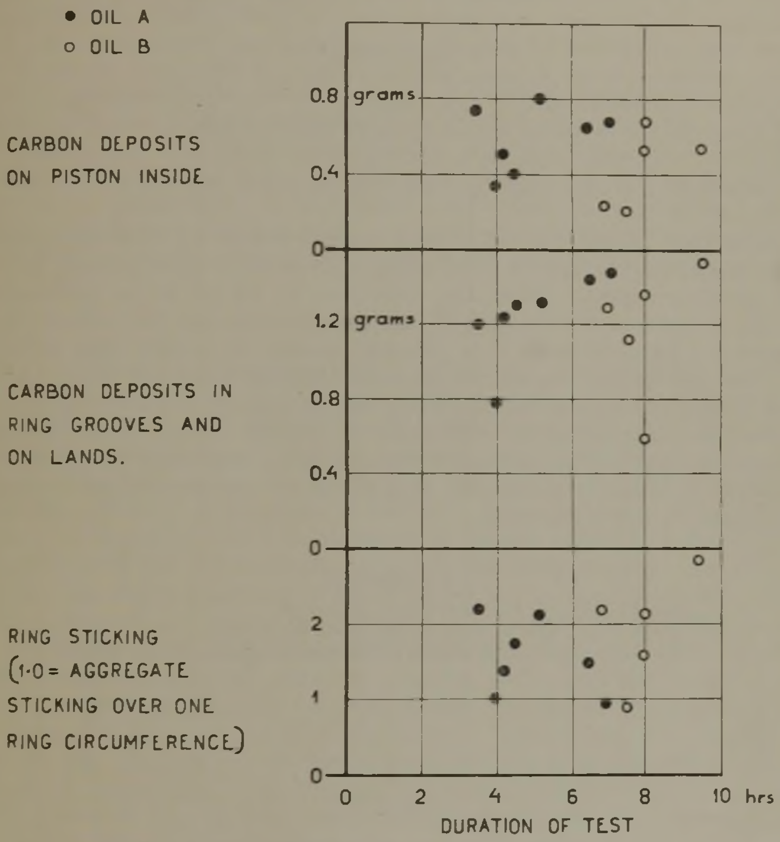

Fig. 4.

CASE OF POOR REPRODUCIBILITY OF RING-STICKING TESTS ON DIESEL ENGINE WITH ETHYLENE GLYCOL COOLING.

Examples covering the degrees of reproducibility of engine-fouling tests are given in Figs. 3 and 4. Fig. 3 represents the amount of lacquer deposit scraped off from the piston and liner surface of a 50-h.p. air-injection diesel engine for a number of nine-hour tests at 15 per cent. load and 44-atm. air-injection pressure. Various tests were carried out with three different lubricating oils; each horizontal part of the stepped line represents the result of a single test. The reproducibility of these tests is very reasonable, and the average differences between the three lubricating oils A, B, and C are only small. This result tallies with the fact that the lacquer deposits referred to are due to incomplete combustion of the fuel.

Another example is shown in Fig. 4, where the results are given of ring-sticking tests with a small pre-combustion chamber diesel engine, running under high-temperature conditions (ethylene glycol cooling). The reproducibility of these tests was rather poor, but seeing that relatively many tests were carried out on each oil, a conclusion can be reached—viz., that oil B is superior to oil A, since, on the average, it allows the engine to run longer for a given amount of ring-sticking. Under such circumstances the only way seems to be to conduct many experiments, in order to obtain statistical data, which enable a conclusion to be drawn. Fortunately the reproducibility of ring-sticking tests is often much better than in the case of Fig. 4.

As the test results depend to such a great extent on mechanical conditions of the engine, engine lubricating-oil tests should, in general, be of a comparative nature. Thus, the behaviour of an oil to be investigated should be compared with that of another oil which has been adopted as a standard. In most cases it is precisely comparative tests with different oils that are wanted, especially when the effect of dopes has to be studied.

Although the testing of lubricating oils in engines is difficult and costs a great deal of time, it has nevertheless to be done in view of the necessity of developing oils with improved characteristics, especially as regards the tendency to cause ring-sticking, and the utter impossibility of achieving the results by any other means.

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PISTON AND PISTON-RING TEMPERATURES *

By P. V. Keyser,† Jr., and E. F. Miller.†

Synopsis.

Piston deposits—Methods of measuring piston temperature—Cylinder wall temperature measurement—Laboratory experiments—Effect of design on piston temperature.

Introduction.

The increase in the specific output of internal-combustion engines in recent years has brought about a number of lubrication problems. Figuring prominently among these is the problem of piston lubrication, which has resulted from the excessive piston temperatures occurring in the piston-ring zone, particularly in high-speed automotive diesel engines. Many of the lubrication practices which are entirely satisfactory in large slow-speed diesel engines are not applicable to the present high-speed types.

Piston Deposits.

The problem of developing an oil which will permit engine operation at piston temperatures of the order of 450° F. in the ring zone is a problem of limiting the extent and nature of carbon formation. The importance of piston temperature might be summed up generally by stating that piston deposits are for the most part the result of some temperature condition existing at the point of deposit. Fig. 8 is presented to indicate the types of deposit likely to be found at various points of a diesel piston together with the approximate corresponding temperatures. On the top land of the piston it is usual to find a rather hard deposit of carbon, which will be formed in a region of temperature of about 550° F. This deposit is sometimes abrasive, and may lead to various degrees of scuffing of the piston and cylinder wall, depending on the load conditions. The deposit found in the piston-ring grooves is a rather dry carbon ranging from soft to hard, depending on the type of oil used. This deposit will be heaviest in the top ring groove, decreasing amounts being found as the oil-ring groove is approached. The temperature range from the top to the third-ring groove is usually from about 450° F. to 350° F. The accumulation of carbon behind the top piston ring can cause abnormally high ring-wall pressures leading to scuffing of the piston ring and cylinder wall. Sludge is invariably deposited in the oil-ring groove in amounts varying with the degree of instability of the lubricating oil and the incompleteness of combustion. The upper part of the piston skirt usually exhibits a varnish-like deposit, the tenacity of which is a function of the type of oil used. This deposit is heaviest on the skirt areas closest to the piston pin, where the pressure of the piston against the cylinder is not sufficient to keep the surface wiped clean. A similar but lighter varnish frequently forms on

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† Socony-Vacuum Oil Company, Inc.

the piston-pin bearings. In the piston-pin relief areas and also on other parts of the skirt an oily soot is often encountered. It is believed that this deposit consists mainly of products of incomplete combustion of fuel and lubricating oil that are scraped down the cylinder wall by the piston rings. It is this sooty formation which, on reaching the crankcase, causes the blackening of the lubricating oil. This is a characteristic which is typical of some high-speed diesel engines.

The designer of a piston is faced with the problem of temperature control. If he chooses to use aluminum instead of iron or steel, in order to make use of the greater heat conductivity of aluminum, he must provide sufficient clearance between the piston and cylinder wall when the engine is cold to forestall the possibility of piston seizure when the engine is run under heavy load. There is the problem of providing sufficient strength when the piston operates at high temperature and pressure due to heavy load. The designer must consider the possibility of localized heating, which can cause piston distortion, and, if great enough, can cause piston fracture because of unequal expansion. Generally, then, it might be said that the maximum continuous operating load of an engine is limited largely by the maximum piston temperature which will allow satisfactory lubrication. Since supercharging provides an effective method of increasing the output of an engine, it appears that it can be done only if a satisfactory means is employed for limiting the maximum piston temperature.

METHODS OF MEASURING PISTON TEMPERATURE.

In the course of the lubricating-oil development work at the Socony-Vacuum Oil Company's laboratories, it has been found useful to make a study of piston temperatures. Most of the work has centred about the temperature studies in connection with the lubrication of diesel engines. A short investigation has also been made of the temperatures occurring in a small single-cylinder gasoline engine.

There are several methods which can be used for measuring piston temperature. One system that is sometimes used is that of the continuous-contact type, in which a thermocouple is embedded in the piston at the desired point and led by a flexible wire, made up of iron and constantan, along suitable supports to a point outside the engine. This system, although giving reliable results, can hardly be used on engines which operate at speeds approaching 2000 r.p.m. Another method of piston-temperature measurement is one in which fusible plugs are used. Alloys of known melting points are securely placed in holes drilled in the piston. The engine is then run at the desired set of conditions until the operating temperatures become stabilized, whereupon the engine is shut down and the piston inspected. By noting which of the alloys have melted, one can determine the piston temperature. This method presents the serious disadvantage that only one set of temperatures can be determined at one time, after which the engine must be taken apart and inspected

A third system, and the one which was used in most of our tests, is the intermittent contact thermocouple type. This method was suggested to us by the General Motors Corporation. A diagrammatic sketch of the system is presented in Fig. 1. The thermocouple in the piston is connected to contacts at the bottom of the piston. These contacts mate with spring-loaded contacts attached to a bracket on the liner. The contacts in each case are made of the same metal as is the leg of the thermocouple it connects. The springs loading the movable contacts are of spring steel, and as there is little or no temperature drop across the spring, it is believed that no error is incurred through the insertion of the third metal. It will be noted from the drawing that lava tubing insulates the wire in the holes drilled in the piston. The wires emerging from the lava tubing are then

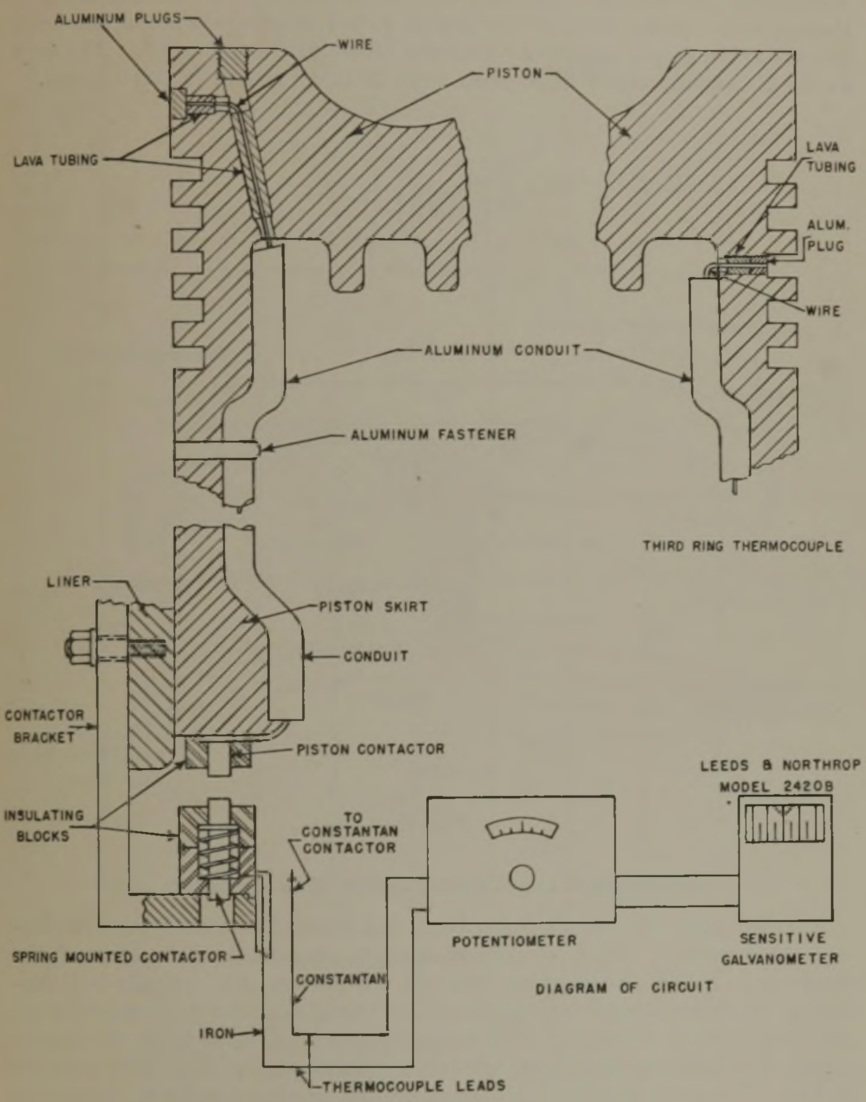

Fig. 1.

wrapped with mica tape and inserted in aluminum conduit. The conduit is attached to the piston in several places to prevent vibration. The wires are soldered to the contacts on the piston. Likewise, the wires connecting the potentiometer to the movable contacts are soldered to the springs. A duration of contact of 35° of crankshaft angle has been found to give ample sensitivity with the instruments employed. As many as three thermocouples have been used at one time with this system.

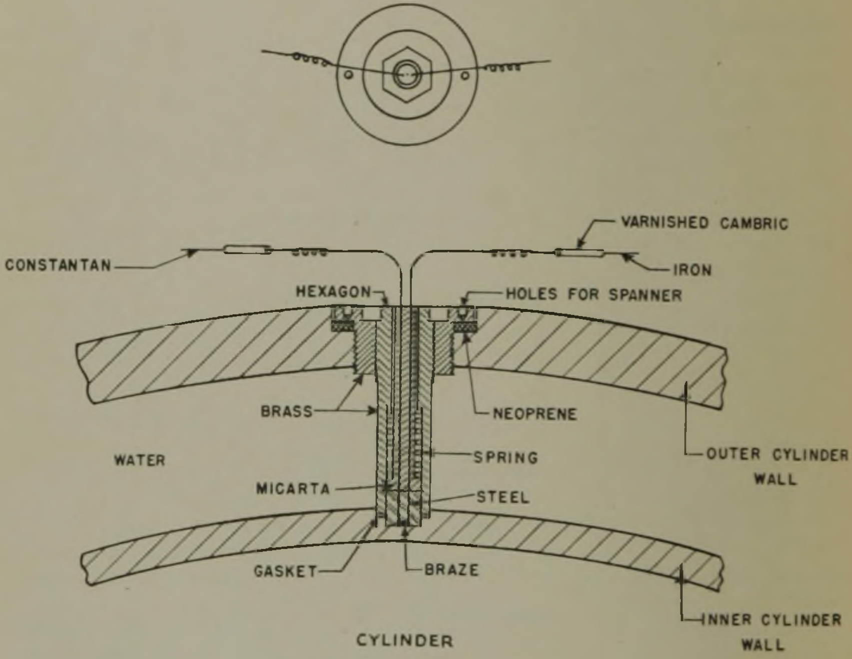

Fig. 2.

Cylinder-Wall Temperature Measurement.

Another useful tool in the laboratory is a method of measuring cylinder-wall temperature. Such measurements give a reliable indication of the lubrication conditions existing between the piston and cylinder-wall surfaces. A thermocouple placed near the inner cylinder wall provides an effective method of making such measurements. Several types of thermocouple installations have been tried in our laboratory. The design shown in Fig. 2 has given excellent performance, responding rapidly to changes in engine operating conditions. Care must be exercised in the installation of the thermocouple to prevent cylinder-wall distortion. Several distances of the thermocouple from the inner wall were tried, including the embodying of the thermocouple flush with the inner wall. It was found that a distance of 0·040 in. from the inner wall gave, as far as we could determine, a thermocouple that was just as sensitive as the one which was flush with the inner

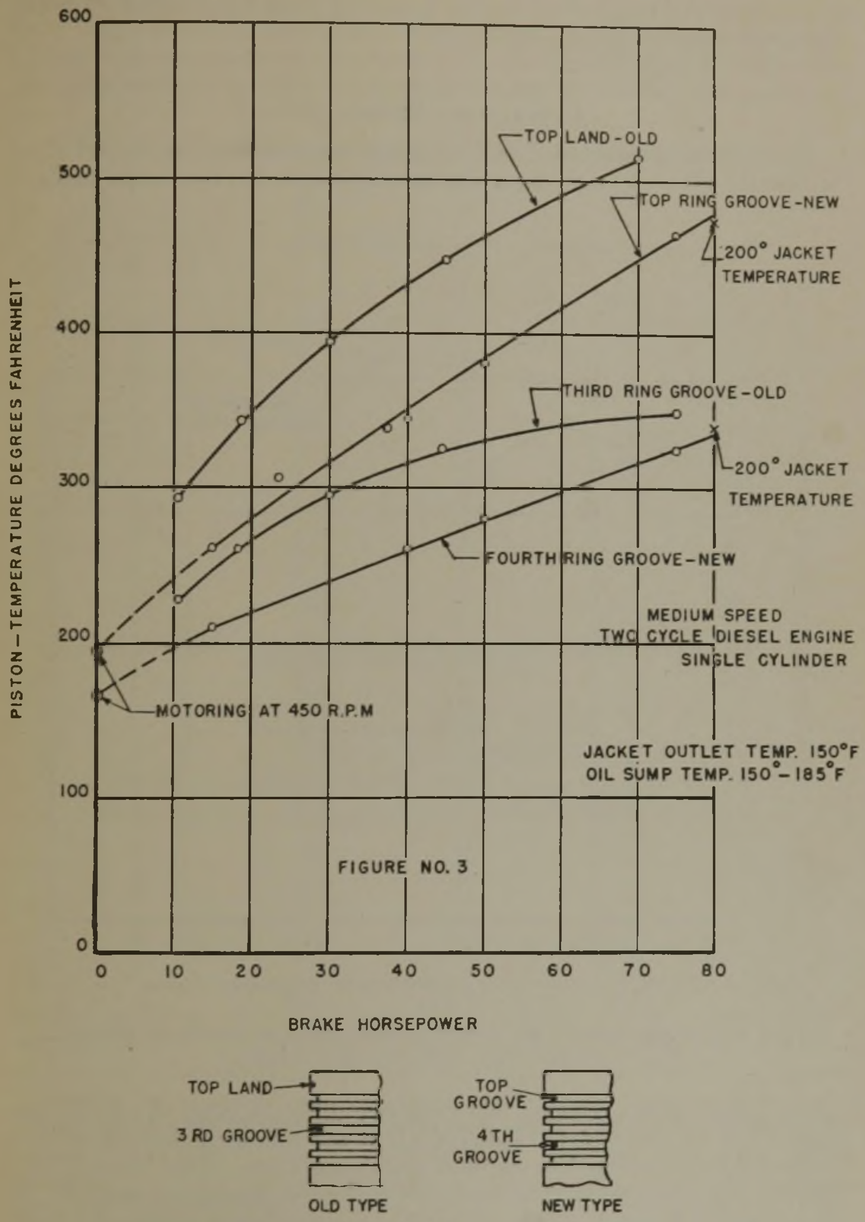

Fig. 3.

EFFECT OF LOAD ON PISTON TEMPERATURE.

wall. This thermocouple was used in an engine equipped with a cylinder liner of the wet type. The design, however, could be modified to permit use with other types of cylinders.

LABORATORY EXPERIMENTS.

Much of the experimental work on piston temperature has centred about measurements made in an experimental single-cylinder, two-cycle, medium-speed diesel engine. During this work two different types of pistons were used. The piston called " Old Type " in the following discussion has a waffled inner crown and is made of aluminum alloy. The " New Type," also of aluminum alloy, has many deep ribs on the piston interior. In both cases oil is sprayed from the top of the connecting-rod on the under side of the head of the piston. The New Type piston is fitted with a metal baffle which traps oil in the interior of the piston for the purpose of cooling.

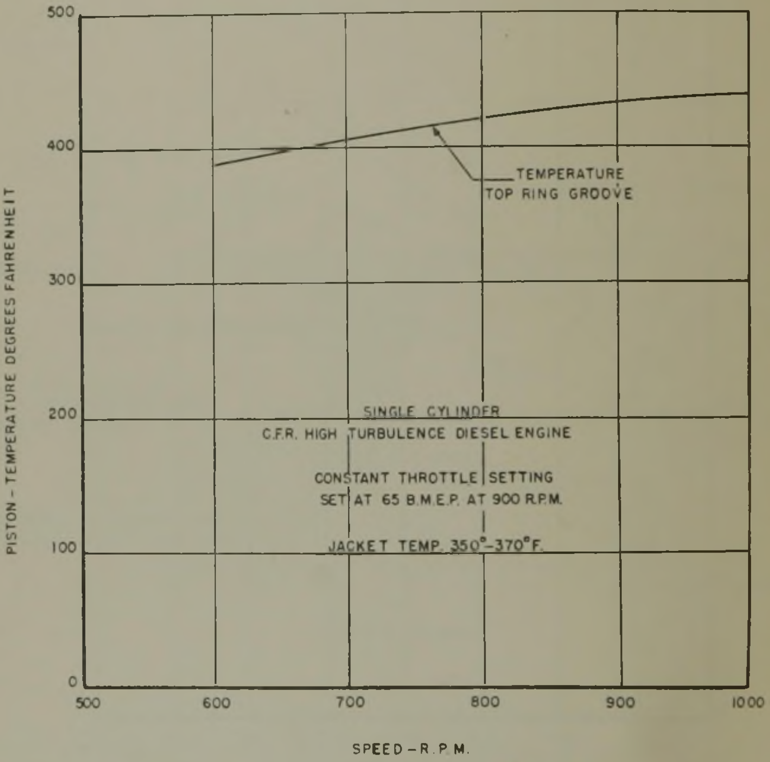

FIG. 4.

EFFECT OF ENGINE SPEED UPON PISTON TEMPERATURE AT CONSTANT THROTTLE SETTING.

The intermittent-contact type of thermocouple described above was used in the piston-temperature measurements made in this engine. The results are summarized in the form of curves presented in Fig. 3. These curves show the effect of horse-power output on piston-ring groove temperature. Both speed and throttle position were varied in these tests, the lower horse-powers naturally being obtained at the lower engine speeds. The curves show that any oil which reaches the top piston-ring groove will be subjected to a temperature of 440° F. (227° C.) when the engine is operated at 75 h.p. The effect of speed on piston temperature at constant horse-power output is shown in Table I. It appears that when the engine was operated at constant horse-power at low outputs, there was a small increase in piston temperature with speed. When the engine was operated at 40 h.p. there was practically no change in temperature with speed.

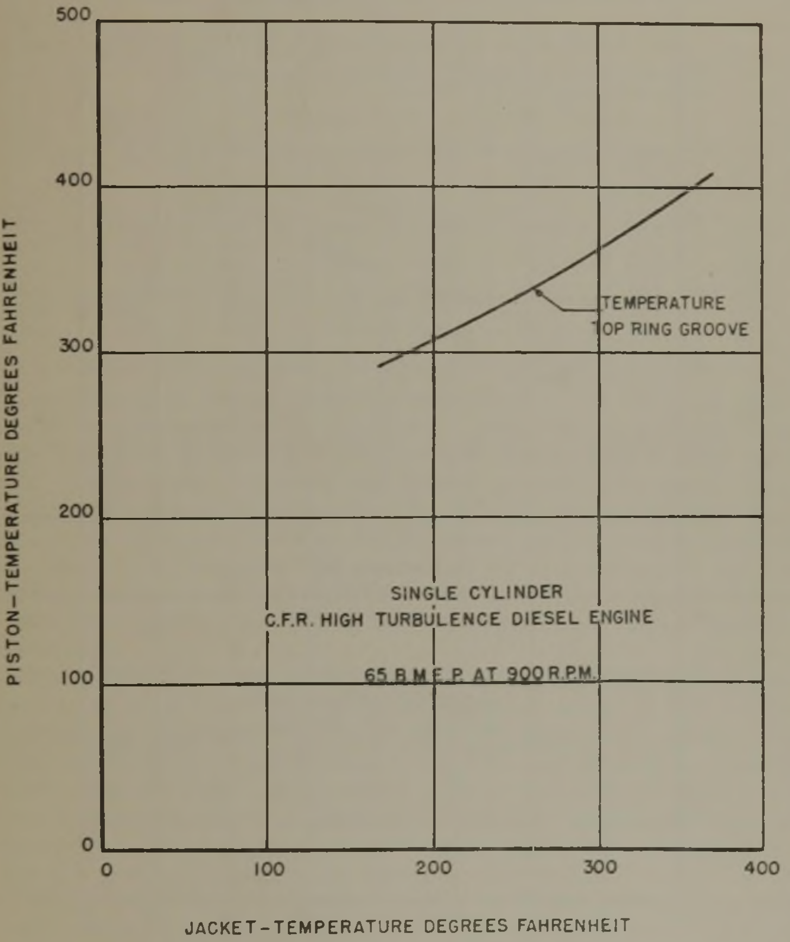

FIG. 5.

EFFECT OF COOLING JACKET TEMPERATURE UPON PISTON TEMPERATURE AT CONSTANT LOAD.

Piston-temperature measurements were made on a Co-operative Fuel Research High-turbulence Single-Cylinder Diesel Engine by the intermittent contact method. The engine was fitted with an aluminum piston for this work. Two curves are presented, Fig. 4 showing the effect of load on piston temperature at constant throttle setting, and Fig. 5 showing the effect of jacket temperature on piston temperature. In both tests ethylene glycol was the jacket coolant. From Fig. 5 it appears that a decrease of 100° F. in jacket temperature will bring about a reduction in piston temperature of about 50° F.

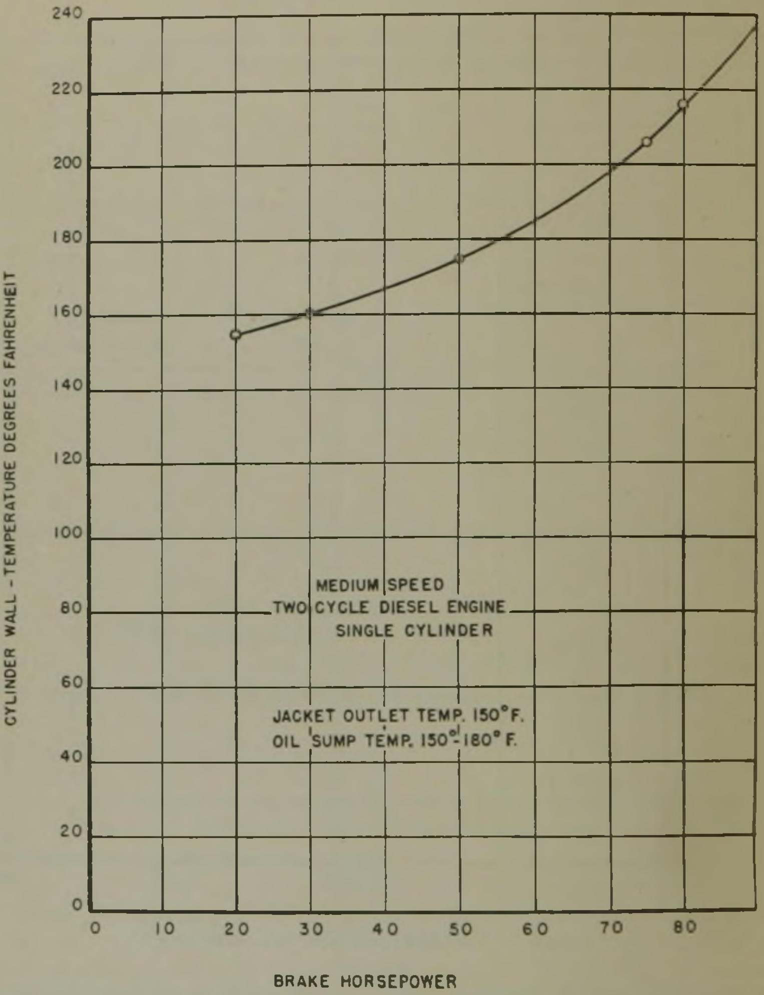

Fig. 6.

EFFECT OF LOAD ON CYLINDER-WALL TEMPERATURE.

The effect of horse-power output on cylinder-wall temperature was investigated in the medium-speed two-cycle engine. These results are plotted in Fig. 6. In this test both speed and load were varied keeping the water outlet temperature constant at 150° F. The changes in cylinder-wall temperature, therefore, are the result of changes in both load and piston speed.

TABLE I.

Effect of Speed on Piston Temperature at Constant B.H.P.

r.p.m.	Beam Load, lb.	b.h.p.	Temperature Degrees Fahrenheit				
			Jacket	Oil	Cylinder wall	Top ring groove	Fourth ring groove
433	102	14·7	148	130	160	250	205
752	60	15·1	150	165	165	295	230
580	152	29·4	150	155	160	305	225
746	119	29·6	151	160	170	330	245
594	202	40·0	150	157	170	345	255
754	159	40·0	150	170	170	345	260

It was found on several occasions that when a new liner and piston assembly were installed in the engine and a heavy load was placed on the engine before the piston rings were properly seated, that blowby would result. Under these conditions the blowby gave an immediate rise in cylinder-wall temperature as indicated by the cylinder-wall thermocouple. The same condition resulted when blowby caused by ring sticking occurred. The cylinder-wall thermocouple connected to a temperature recorder provides a useful tool, especially on this a two-cycle engine where a quantitative measure of blowby is impossible because of the ports in the cylinder liner.

Piston-temperature measurements have been made in a single-cylinder, four-cycle, air-cooled gasoline engine, having a 2⅛-in. bore and 1¾-in. stroke. The fins on the air-cooled cylinder are spaced to about a ½-in. pitch. Fig. 7 shows the variation of piston temperature with load at constant speed as determined by the fusible-plug method. These tests indicate that in this engine the second piston-ring groove operates at a temperature approximating to that of the top ring groove. It will be noted that the temperature of the top piston-ring groove in the air-cooled engine is about 440° F. (227° C.), or about the same as the temperature of the top ring groove in the medium-speed two-cycle engine. A rough indication can be obtained from this comparison of the price which must be paid for such air-cooling of engines, for here we observe that although the air-cooled engine is operating at an output of only 36 lb./sq. in. B.M.E.P., the piston temperature is just as high as in the water-cooled, two-cycle engine when operated at a B.M.E.P. of about 80 lb. per square inch.

EFFECT OF DESIGN ON PISTON TEMPERATURE.

The heat in the piston-ring zone is dissipated in several ways. A considerable amount is conducted away from the piston through the piston rings which are pressing against the relatively cool cylinder wall. Some heat will be taken up by the lubricating oil which comes in contact with the inner crown of the piston. Heat is conducted to the piston skirt, which in turn transmits the heat to the cylinder wall. A small portion of the heat is carried through the wrist-pin to the connecting-rod, and then dissipated to the crankshaft. The amount of heat thus conducted has been known, in unusual cases, to cause the bearing temperature to be sufficiently high as to necessitate a change in design. A heat groove above the top piston-ring groove is sometimes employed in an endeavour to

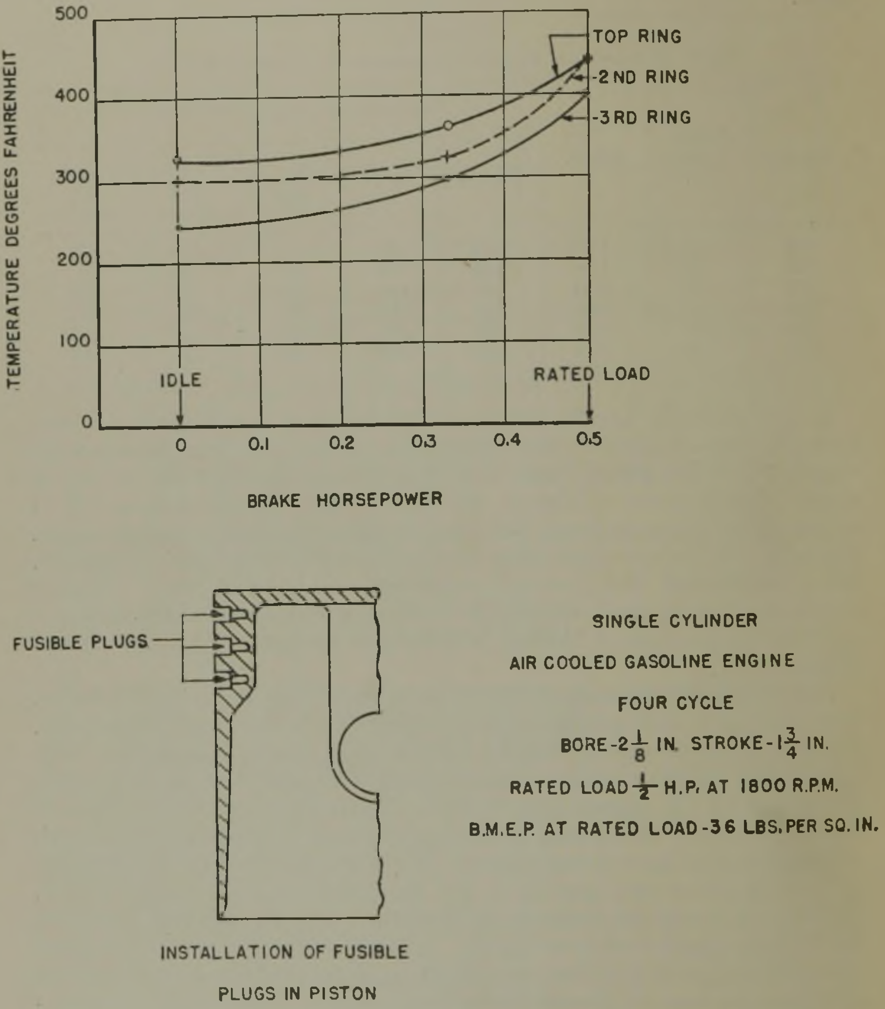

Fig. 7.

EFFECT OF LOAD ON PISTON TEMPERATURE AT CONSTANT SPEED.

prevent the heat from the head of the piston from being transferred to the immediate vicinity of the top-ring groove. Instead the heat tends to be distributed more uniformly over all the piston-ring grooves. Too much dependence cannot be placed on this method of controlling piston temperature, for the effectiveness of the heat groove is probably reduced with service because of carbonization. A method of limiting the amount of heat reaching the piston-ring zone is that in which the path of the heat from the head of the piston is restricted by making the piston material above the top piston-ring groove very thin, and to use oil sprayed from the top of the connecting-rod for cooling purposes. Pistons of this type are usually made of iron or steel, for it is desired to restrict the flow of heat as much as possible. Also, it is necessary to use a strong material because of the thinness of the section connecting the piston-ring region with the piston head. In engines employing an ante-chamber to control the combustion process, the practice of directing the throat of the ante-chamber toward the piston causes localized heating, sometimes sufficient to cause ring-sticking at the latter point.

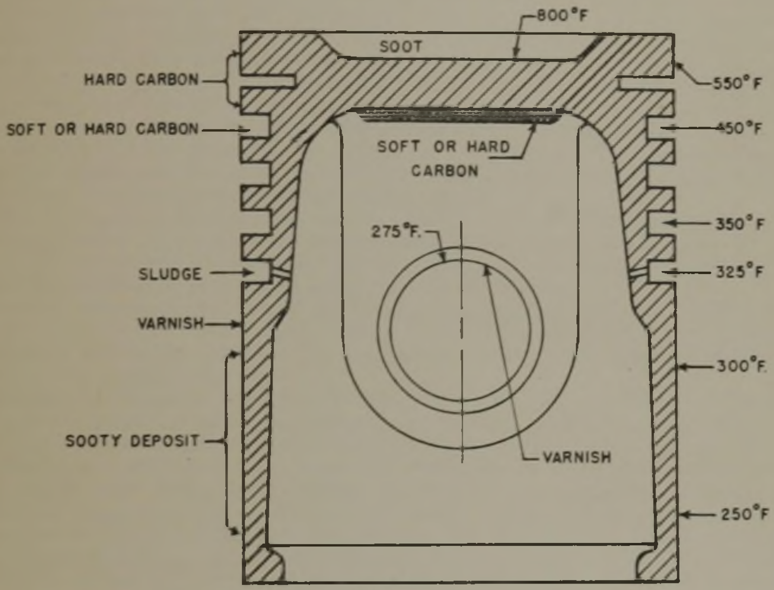

Fig. 8.

NATURE OF PISTON DEPOSITS AND CORRESPONDING PISTON TEMPERATURES.

It must not be inferred from the above discussion that it is desired to have the piston run as cool as possible, for, assuming that the temperature of the piston could be reduced to some very low figure—say 150° F.—to do so would handicap the combustion process by increasing the delay period and promoting incomplete combustion. In this connectiom it should be mentioned that pistons are sometimes cupped at the top so as to form a large percentage of the area of the combustion chamber. This is done to assist combustion of the fuel, the hot surface of the piston taking the place of the comparatively cool cylinder wall. At first thought one might think that the designer of a piston must choose a temperature at which he desires the piston to operate, and then design the piston accordingly. This is hardly the case, however, for the designer will have all he can do to bring the piston temperature down to a point where an engine may be operated at high output without resulting in severe punishment of the lubricating oil.

Morning Session B, Wednesday, May 24th.

Papers on Testing of Lubricants.

REPORTER'S SUMMARY OF PAPERS.

(Mr. E. A. Evans, M.A.I.E., F.Inst.Pet.)

Mr. E. A. Evans, in summarizing the Papers on Testing of Lubricants presented at the Morning Session on May 24th, said that lopsidedness was not a word which came easily to the mouth of an Englishman, but Bouman used it in his papers with emphasis. It was descriptive, and produced a picture of errors which the Institute of Petroleum was anxious to eliminate. A lopsided conception of lubrication was fatal to progressive thought. In the past much time had been devoted to chemical and physical testing of lubricants, and still the struggle went on; they were ever hopeful of achieving a useful result. Throughout that struggle there had been a consciousness that mechanical testing might be introduced to short-circuit the would-be academic attack. Many years ago Thurston designed a friction testing machine to study oiliness. Improvements and modifications had appeared and faded away, leaving behind much the same speculation as that which had preceded them. Relatively slow speeds and low pressures did not present a real lubrication difficulty, but when speeds began to increase, engine power shot up, and the whole train of engineering progress became alarmingly dependent on lubricants and lubrication.

New methods for development were necessary. It was no longer safe to take a chance, fortified only with some physical measurement such as viscosity. It certainly was not economic to await the laborious findings of a full-scale engine or machine test. Dimensional or mechanical testing became imperative.

At that time mechanical testing of lubricants was standard practice. The objective of the papers in this section was to survey mechanical testing with a view to standardization, and so to eliminate any lopsidedness which might exist in appraising lubricants for specific purposes.

Bouman had waged a minor war, in the past, on the supposed value of oxidation tests as the criteria of oil decomposition in an engine. He had returned to the fray with more ammunition to defend his front. Nobody would disagree with him in his suggestion that the changes which occurred within the oil in an internal-combustion engine were very complicated; likewise there would be agreement on his remark that there was a variety of conditions in the engine which would produce a variation of reactions. It was too much to hope that one could reproduce all those conditions on a laboratory bench. It was much simpler and more reliable to resort to mechanical testing in an engine. Bouman had shown that, with the aid of an engine, it was possible to give a quality rating for oil. The three oils which he used were placed in their respective positions according to the amount of carbon in the ring grooves, but when they were rated according to the weight of carbon on the piston crown, the order was reversed.

Bouman was not so bold as to condemn an oxidation test. He admitted that it had a limited sphere of usefulness, and referred to the so-called Delft " baking " test, developed in his own laboratory. This test seemed to be almost identical with that described to the Institute by Hoblyn in 1925. Bouman was not the man to introduce an air of finality into his paper; on the contrary, he had reopened an old controversy. He was alive to all the difficulties encountered in engine testing, but however great they might be, they must not eclipse the desire to introduce engine testing into a research problem. He cited results, obtained when using a compression-ignition engine, which went to show that the study of the lubricating oil was insufficient without due regard to the fuel influence. None of those results could be obtained by any other means than engine testing.

The paper by McNicol, Williams, and Lamarque formed a pleasant link with Bouman's. They did not propose a new test, but modestly described an experimental technique extremely simple in character, but full of possibility. The extensive facilities which they possessed at the Research Laboratory of the Institution of Automobile Engineers led one to postulate that engine tests would be made, to prove or disprove the practical value of the test for the ring-sticking properties of lubricants. Before leaving the subject, it would be interesting to know whether ring-sticking was a common phenomenon with low-viscosity solvent oils.

So much uncertainty existed about the evaluation of extreme pressure lubricants that another opportunity to consider the subject was welcomed. There was no universally accepted test machine. It was probable that standardization could not be effected until much more was known about film rupture strength and the influence of surface finish.

Clayton's paper at the General Discussion on Lubrication arranged by the Institution of Mechanical Engineers in 1937 did not create the interest which it warranted, possibly because the Four Ball Machine was not widely known except by name. To follow his present paper it might be necessary to be reminded that three ½-in. steel balls were placed in a small oil-bath, and a fourth ball was superimposed so that it was in contact with the other three balls. The top ball was rotated at 1500 r.p.m. The great feature of the machine was that four balls were in point contact, and as soon as a load was applied, boundary conditions existed. When a load was applied there was obviously some slight deformation of the balls, so, instead of having point contact, there was a slight depression, which could be calculated according to the Hertz theory. Any depression larger than that must be due to wear. At a certain load the oil film broke down and the balls seized. The seizure might be so drastic that the balls welded together. On the other hand, the seizure might be slight, and only noticed by a sudden rise in friction. At the moment of seizure rapid wear occurred, the impression was increased in size, and the friction diminished. The lubricant had, in fact, effected a recovery.

In making a test all that one had to do was to put a load on to the lever and run for one minute after connecting the oil-bath to a revolving drum which measured friction or torque. At the end of the run the diameter of the impression was measured and plotted against load. From a series of runs a graph could be drawn. When a seizure occurred, a rapid increase in the size of the impression resulted, which was depicted in Fig. 2. The significance of that was merely that it indicated the load at which breakdown took place, quite irrespective of time. The time to seizure against load was of importance only if it could be correlated with practice. It had been found that the load which caused seizure in 2½ secs. gave an indication of the suitability of the lubricant for a hypoid gear. The friction-time chart showed the recovery time. An oil which seized but recovered quickly was better than one which had a long recovery time. An oil which did not seize was the ideal, provided it did not possess the other features which condemned it.

Oil *E*, containing 10 per cent. lead soap and 2¼ per cent. sulphur, gave no breakdown as shown in Fig. 4*b*. Oil *G*, containing 4½ per cent. lead soap, gave similar results to oil *E*, except that at 150 kg. wear rose to almost double that of oil *E*. The increase in wear appeared to be due to the smaller content of lead soap. Oil *F*, containing 13½ per cent. fatty oil, 1½ per cent. sulphur, and 10 per cent. chlorinated product (3 per cent. chlorine) was described by the makers as " mild," yet behaved as oil *E*, and better than oil *G*. Evidently the improvement was due to the chlorinated product.

Finally, Clayton gave the results of tests on several mineral oils, and came to the conclusion that solvent refined oils were inferior to conventionally refined oils in film rupture strength. That suggestion was too sweeping, because it was not confined to one solvent or one type of oil, nor to any particular degree of refining.

Assuming that a hypoid gear acquired a " surface " during its early life in an E.P. lubricant, Baxter, Snow, and Pierce studied the pre-treatment of the test-pieces in E.P. test machines. Their paper gave sufficient data to prove that pre-treatment gave rise to marked increases in load-carrying capacity to both doped and undoped oils. That was most important, therefore the paper should commend itself to motor-car manufacturers.

The term " dope " was so loosely used at that time that it might very well give rise to a wrong impression. Surely it was preferable to refer to an E.P. addition agent.

So far consideration had only been given to E.P. oils. Evans introduced E.P. greases. Admittedly at the present time they were mainly used in large roller bearings, but when they once became established their use would obviously be extended. They differed from oils in that an E.P. addition agent was insufficient to produce E.P. properties. As Evans was one of the very few people who had given serious consideration to E.P. greases, his paper should be studied in its entirety.

Grease had been improved in recent years mainly under the compulsion of the anti-friction-bearing makers. It was a mistake to think that one grease was suitable for all makes of ball or roller bearings. Evans made a very strong plea in favour of certain lime-soap greases. He was not ready to accept any type of lime-soap grease. In fact, he had laid down a very rigid specification. It was probable that it was very largely through his efforts that grease-makers had improved the stability of greases. Grease-makers might not like to acknowledge that they had received an ultimatum, but it must be acknowledged that the Timken stability test had become both popular and important. The arguments of Evans in favour of the lime-soap greases were clearly set out in that paper. He stated that it might not be common knowledge that although the melting point of the soda grease was higher than that of the lime-soap grease, the consistency of each at temperatures of 140° F. and above was approximately the same, making allowances, of course, for the consistency at atmospheric temperature. The relative merits of lime and soda-soap greases for anti-friction bearings made a very controversial subject. Perhaps, however, the subject was not quite so controversial as it might appear at first sight. Evans told us that he preferred a medium viscosity oil in the grease for the Timken bearing. The Hoffmann Manufacturing Company generally preferred a lower viscosity oil. That difference of opinion was not based on some theoretical consideration, but was actually due to the difference in the design of the bearings. Perhaps the discussion might reveal points of view which might help to clarify some of those apparent anomalies. If there should not be an opportunity for clarifying them at the meeting, there would be facilities for further discussion at the Lubrication Group Meeting which had been arranged to take place in October.

The Timken machine was used by Evans for measuring the extreme pressure characteristics of both oils and greases. That machine appeared to have suffered the same fate as many other allied machines in the U.S.A. Whether some of those machines would be restored to favour, however, now that the mild E.P. lubricant was being reinstated, it was difficult to tell. For E.P. greases, however, the Timken machine did not appear to have a serious rival. It was interesting to note that when using that machine there was some support for the suggestion made by Baxter, Snow, and Pierce, that oils containing chlorine compounds show less wear than those which contain sulphur. Another very interesting point which was emphasized was the effect of finish on film strength. The importance of that could not be overstated, but whether it was cheaper to produce an ideal finish in the engineering shop, or in the back axle with the aid of an E.P. lubricant, must be left to experts in costing. The scientific mind of the engineer would no doubt prefer the result of his own art, whereas that of the chemist might favour the physico-chemical reactions between active compounds in the lubricant and the metal surfaces.

Were they really safe in coming to definite conclusions about the properties of chlorine when only one or two chlorine compounds had been tried?

If they accepted the authors' views, it appeared that they had learned that :—

(1) Mechanical testing was essential;
(2) Oxidation tests were misleading;
(3) Chlorine compounds were serious competitors to sulphur compounds;
(4) Pre-treatment of gears was a possibility;
(5) Surface finish might recast views on E.P. lubricants.

DISCUSSION.

DR. K. O. MÜLLER said that he wished to express his thanks for the opportunity to take part in the Summer Meeting, and he hoped that his English would be excused.

He had read the papers presented at the various sessions with great interest and appreciated the progress made during the last few years.

The Chairman's request that he should open the discussion was accepted as an honour which he was pleased to undertake.

With reference to motor oils, he still felt that more importance should be attached to the viscosity index or pole-height in connection with easy start at low temperatures. With reference to the physical and chemical characteristics to be determined in the laboratory, his view was that alteration should first be devoted to the behaviour of oils in engines working under practical conditions; then from this a laboratory method should be devised—such as ageing tests—which would reproduce as closely as possible the alteration of oils in engines. An attack on those problems in that manner would show that physical data such as flash-point and laboratory pour point could be disregarded, as they had no bearing on practical conditions. They should, for example, be usefully replaced by viscosity measurements at low temperature and volatility.

He would also like to emphasize that lubricating oils should not always be blamed for unfavourable engine performance, for in his view the latter depended on the fuels, engine design, and metallurgy.

With regard to E.P. lubricants, he had experimented for many years with different testing machines such as the Timken 4-Ball, Almen, and S.A.E. types, but he had never found them to agree with each other, and their results did not, in his opinion, have any relation to practical operation.

At the same time, he would say that he had never been able to understand why those E.P. lubricants should ever have been needed. By that he meant, whilst the lubricants might be essential for certain designs of gears, those gears in themselves were really not essential, and it seemed to him that they existed solely for reasons of fashion. In other words, it was, as far as he believed, possible to give the vehicle the necessary free height above the road with worm-gears which could be so designed that their lubrication was provided by simple oils, and this dispensed with the need for complex lubricants having chemical corrosive effects.

The conditions for lubrication were obviously simpler if ordinary gears were robustly designed, provided adequate workmanship was put into the gears, and surface finish was carefully attended to. The preparation of surfaces would open up a new outlook, and deserved more attention.

Mr. C. H. Barton said that in the absence of Mr. Bouman, owing to military duties, he had been asked to say a few words about his paper. Mr. Bouman had emphasized two problems which were well known to all who were concerned with engine tests on lubricants. Those were the difficulty of obtaining reproducibility of observations and the variability of results with change of working conditions. The latter phenomenon gave rise to uncertainty in the interpretation of results, especially when data from one engine were applied to another. The reproducibility of results under constant test conditions was a necessary criterion of the validity of the observations.

With regard to ring-sticking at high cylinder temperatures in gasoline engines, he noticed that Messrs. McNicol, Williams, and Lamarque's heating test indicated that the tendency of a lubricating oil to give sticking increased with the volatility of the oil. In the course of the high-temperature ring-sticking experiments with engines, with which he had been concerned for about ten years, it had been found that the least volatile mineral oils—*e.g.*, bright stocks—were not outstandingly resistant to ring-sticking, as would be expected from their low volatility on the basis of Mr. Williams' results.

Of the various factors which controlled ring-sticking at high temperatures, the metals involved did not appear to exercise a marked influence. Thus, chromium-plated and nitrided cylinder liners gave about the same rapidity of ring-sticking as that with cast iron. Tinned rings also were comparable with cast iron ones in their behaviour under sticking conditions. He had also tried the addition of 0·5–1·0 per cent. nitrogen peroxide in the air/fuel mixture by dissolving the appropriate proportion of that compound in gasoline, but no effect on the rate of ring-sticking was found (cf. Hanson and Egerton, *Proc. Roy. Soc.*, Series A, **163**, p. 90, 1937).

The question of "varnish" deposition on the pistons of spark-ignited engines had been referred to in one or two of the papers at the meeting. He had had the opportunity of discussing that matter with several authorities when he was in America six months ago. The one point on which they seemed to agree was that the ill-effects of varnish occurred only during the early life of an engine, when piston-cylinder clearances were at a minimum, in causing the pistons to tighten up after the engine had been shut down. Some people considered that the varnish effect was greatest at low oil temperatures and with less chemically stable oils; and others that high oil temperatures were required to produce objectionable amounts of varnish. The number of instances in which varnish had actually caused running trouble was relatively few, and had been exaggerated by undue publicity.

Mr. J. L. Taylor said that he was glad Dr. Pye had invited comments on the question of reproducibility when the engine was used as a testing unit, since it was well known that reproducibility was not easy.

He would also remind members that, in addition, engine tests were costly and laborious, so that cases could and did arise where engine design changed so rapidly that engine tests lost a great deal of their value by not being completed before the design became obsolete. He wished to stress, therefore, the necessity of endeavouring to obtain the maximum amount of information from a laboratory test, preferably one which was already well established, such as the British Air Ministry oxidation test. He was glad to learn from Dr. Pye that this was still considered a useful test to which to purchase aircraft-engine oils, and thought that its usefulness might be extended in the direction of determining ring-sticking tendencies of oils, and their liability to corrode newer bearing-metals, and also to form lacquer. Some information regarding the first of these properties might be obtained by observation of the occurrence or otherwise of nodules of carbonized or oxidized oil in a ring round the test-tube where the bubbles broke, and regarding the third property by the ease with which the tube could be cleaned by various solvents. As regards the second property, he thought that a determination of acidity, not necessarily total acidity by the conventional I.P.T. method, but rather water-soluble acidity, might be useful. He would like to hear whether other oil chemists had any observations on these suggestions.

There were some comments on one or two of the papers he wished to make. In the paper by Williams and co-workers on the suggested method for determining ring-sticking properties of oils, there was an apparent contradiction in ascribing maximum ring-sticking tendency in one instance to most volatile oils and in another instance to least volatile oils. He noticed, however, that the temperatures were different in the two cases, and thought possibly the test was one which was very sensitive to changes in temperature. As regards the observation that breakaway-force decreased with increasing sample, he thought this was to be expected since the ratio of air to oil was lower and, therefore, the oil would not be oxidized to such a great extent, and, on the other hand, there was more oil to dissolve its own oxidation products.

Lastly, in view of the comments made at the meeting on the effect of surface finish on, for example, "film-strength," he would like to read, for the benefit of those members who were not familiar with it, a part of the review he had made in the Institute's publication "Petroleum Technology in 1938." "A feature of the year's work is the attention given to the effect of the metal surface on wear. Improvements are claimed for surfaces chemically treated or finely finished. In the former case, by chemically treating bearing surfaces so as to dissolve selectively ferrite and other bodies considered as harmful detritus formed by attrition, Foster claims to produce a surface with a bearing capacity six times that of an untreated surface when tested with a straight mineral oil. The surface characteristics of cylinders and pistons should be conducive to polishing by small-sized abraded particles, says Teetor, claiming good results for the Ferrox method of surface treating. In the case of fine finishes papers have appeared by Sherman, working on the effect of surface finish on load carrying capacity, and by Connor, who relates surface roughness and oil consumption using a profilometer to study roughness. Sherman's figures are worth reproducing. Using the S.A.E. machine, he gets a figure of 117 lb. for a surface finish of 16–18 and of 217 lb. for a figure of 1–2 micro inches."

Dr. A. von Philippovich said that it was impossible to get a good correlation between an oxidation test in the laboratory using all kinds of engines and oils, and working under varying conditions. In the D.V.L. much was being done to get repeatable results with engines and good correlation with laboratory tests.

When precautions such as measuring side clearance of piston rings to 0.001 mm., the blow-by from 20 : 20 seconds and so on, were taken, engine tests gave good repeatable figures. Tests on ring-sticking over a range of temperatures showed that there was a minimum time in which rings would stick. This minimum was not at the same temperature for different oils, and the form of the curve was also quite different. That showed that there was only a certain range in which ring-sticking was of importance. Above that range engine failure was due to non-lubrication (that meant volatility and lack of "oiliness"). Below that range sludging would be of greater importance.

In consequence of that, laboratory tests must also take into consideration a wide range of temperatures. Work carried out in France had shown that a coking test at two different temperatures (oxidation at 150° C. and coking at 250–300° C.), when the coking was supposed to take place in the absence of oxygen, gave maxima which could account for the minima of ring-sticking time.

He thought it was worth mentioning that some valuable work on volatility seemed to have been forgotten. F. H. Garner published an article on the subject in *J. Instn Petrol. Tech.*, **7**, 98 (1921), and Hoblyn referred to a method of heating oil in a dish at 250° C. and measuring the volatility and the amount of asphaltic matter produced in *ibid.*, 1925, **11**, 1.

As a result of tests made by the D.V.L. it became apparent that nitrogen oxide had no specific influence in detonating combustion except by increased temperatures as a consequence of knocking.

Mr. H. J. Young said that it might have been overlooked that Robert Sulzer had read a paper on the subject of Piston and Piston Ring Temperatures in 1926, which appeared in Vol. 68 of the *Transactions of the Institute of Naval Architects*. Sulzer dealt with the temperatures, during the various strokes, of the piston, piston rings, and cylinder wall of a two-cycle marine diesel engine. In as far as he (Mr. Young) understood Sulzer's work, it showed that the top ring of the engine attained a maximum temperature lower than that of the top ring of a steam-jacketed steam engine; that the problem of overheating was confined mainly to two areas—namely, the surfaces of the top piston ring grooves and of the upper portions of the cylinder walls. Sulzer found that no thermal fluctuations took place at more than 5 mm. depth below the liner face, but that violent fluctuations occurred close to its working surface. He (Mr. Young) suggested that temperatures occurred which brought in other considerations concerning cylinder wear than those commonly discussed; for example, the effect of certain nascent carbonic oxides on metal momentarily almost molten.

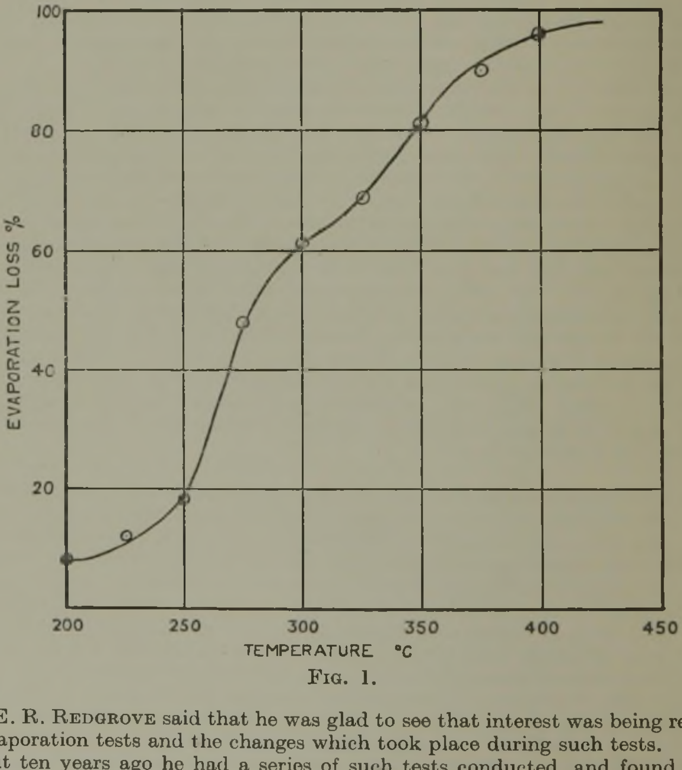

Fig. 1.

Dr. E. R. Redgrove said that he was glad to see that interest was being revived in film evaporation tests and the changes which took place during such tests.

About ten years ago he had a series of such tests conducted, and found that the evaporation curves were far from regular. A typical example was given in Fig. 1.

The "kink" in the curve was common to both distillates and blends of distillates and residual stock; the temperatures between which it occurred varied with the oil itself, the surface-area/volume ratio of the sample tested, and the volume of air passed over the surface of the sample. The treated samples were analysed for "Asphaltene" and "Carbene."

In all cases the "kink" appeared to be associated with serious decomposition of the oil, and it did not seem possible intelligently to interpret the results, which for that reason were not published. At that time ring-sticking and "varnish" or "laquer" were not the serious problem they were to-day, and in view of the present position he thought it might be advisable more closely to investigate the evaporation test over a wider range of temperature.

From the papers before the meeting and from the remarks of various speakers it was evidently appreciated that no one oxidation test could adequately foretell the behaviour of an oil under the varying conditions of temperature, nature, and composition of bearing surfaces and extraneous contamination which it was likely to encounter during service. He thought that nearly all sludging troubles were due to contamination from outside the crankcase, principally caused by incomplete combustion of the fuel, and only very slightly by decomposition of the lubricating oil film around the top rings.

With all those varying factors to contend with, it became necessary to adopt a control test for the chemical stability of a lubricating oil, and he thought the present Air Ministry test as good as any, provided that the asphaltic matter formed after oxidation was also determined and taken into account in assessing the merits of the oil tested.

Mr. J. Romney said that they had heard a great deal at that meeting about laboratory tests and service tests, and suggestions had been made that laboratory oxidation tests were not very useful, and did not accurately forecast the behaviour of oils in service. He would point out that it had been emphasized at that meeting that it was extremely difficult to reproduce engine results in duplicate tests even when all possible precautions were taken to control the conditions. This was not true of laboratory tests, and even complicated oxidation tests were, in general, capable of giving good agreement in duplicate determinations. It seemed reasonable, therefore, to select an oxidation test which was theoretically logical, and if its results could be directly correlated with those of a big series of service tests, to adopt it as a guide. That was what had been done by Mr. Wilford using the Air Ministry test in the laboratory and a very large number of vehicles on the road. He would make it clear, of course, that when he referred to the Air Ministry test, he was thinking of the actual oxidation method. Several people pointed out at yesterday's meeting how much more useful the test could be if asphalt development after oxidation was measured.

The point had been raised at that meeting that the value of the test could be further increased by measuring more properties of the oxidized oil, and the acidity had been mentioned as a probable reflex of the tendency towards corrosion. It might interest the meeting to know that he was concerned in some work on this particular point, and as far as their results went, it was impossible to correlate the acidity of the oil left in the oxidation tube with corrosion as reflected by the loss in weight of test-pieces immersed in the oil.

Mr. J. C. Jennings said he was particularly interested in the ingenious test described by McNicol, Williams, and Lamarque. It was rather difficult to decide to what extent it was a coking test, and to what extent it would be regarded as an oxidation test, but undoubtedly oxidation played an important part. It would be of interest to look into the effect of using different metals, active and inert, as well as glass, used as materials for the dishes, and to note whether the order in which different oils were rated by the test was in any way affected by the metal used. As a preliminary test it would probably be sufficient to compare the results already obtained with those obtainable on the same oils using copper dishes. He found the curve plotting evaporation less against temperature (Fig. 5 of the same paper) rather surprising. It was practically a straight line, with a tendency towards flattening at the higher temperatures. He had had occasion recently to prepare curves of a similar type, but over the range 120–200° C. for a whole series of oils of different types, and these showed a very pronounced steepening at the higher temperatures. It seemed probable that in the case of the dish test, oxidation and polymerization at the higher temperatures were a contributory factor.

Mr. Beale had referred to the almost phenomenal cleanliness and freedom from carbon of a crankcase oil which was being constantly filtered during service. It was difficult to explain the particular case which he quoted, but it was certainly a fact that removal of solid matter during service had an important effect. Some time ago his laboratory carried out a series of tests to study the effect of typical crankcase carbon. A sample of this was filtered off from a used oil and washed free from oil, and very small percentages were introduced into various new oils, which were then compared, on the Air Ministry oxidation test, with the uncontaminated oils. The effect was marked in every case, and quite considerable in some cases. In fact, results like double coke values and asphalt after oxidation were obtained in several cases with quite small percentages, even allowing for the added carbon to give a coke value and asphalt content of 10 per cent. These series of tests, of course, were a practical demonstration of the generally accepted opinion that the degradation of oils was an autocatalytic process, the products of reactions being catalysts for the progressive oxidation of the oil.

Mr. D. Clayton said that the effect of surface finish on the results with the Timken machine as described by Mr. J. H. Evans was very interesting, and supported the claims made by Chrysler for their "superfinish." This effect might explain some of the differences found between different extreme-pressure lubricant testing machines. It was noteworthy, however, that as regards coefficient of friction, and to some extent as regards load carried, all the tests with polished surfaces grouped themselves together and lay apart from those with ground surfaces; this suggested that, provided the surfaces were reasonably well polished, the degree would not be critical. It was thought that the explanation for the change with finish might lie in the formation of partial fluid film between the cup and the block. Support for this was found in Peppler's measurement of fluid film pressure in a machine of the Amsler wear-testing type,* and S. Way's explanation of pitting in a similar machine in terms of the hydrodynamic pressures.† Moreover results of Maag's were quoted on p. 742 showing the effect of viscosity. On the other hand, the impressions were shown to be not greatly different in size, and it would be interesting to know whether the appearance of the scars suggested any difference of régime.

The effect of plating on the behaviour was also interesting. The explanation might be in greater effective smoothness, or in different cohesional properties; little was known on the latter subject. There was a possible link here with the tin-plating of pistons, etc., where a difference of "pick-up" was found. This connection might be important; it was thought that the same fundamental phenomena were involved, viz. the transition from fine to course particle removal.

Messrs. Baxter, Snow, and Pierce also provided a similar link with cylinder operation as regards pre-treatment, as their work was closely related with some of the anti-scuffing treatment of piston-ring surfaces to facilitate running-in.

There was a noteworthy importance in this pre-treatment, because it would allow surfaces to be treated with extreme-pressure dopes which, though effective, could not be marketed on account of other qualities such as odour, instability, etc. On the other hand, as regards comparison with running-in treatments, the pre-treatment was not an alternative in so far as initial roughnesses were removed during running-in.

The small, rough impression found with the Timken machine was similar to that obtained in his (Mr. Clayton's) tests with the 4-ball apparatus when "mild" seizures occurred, and it had been found at one stage in the load range for powerful extreme-pressure lubricants (Fig. 5 (c)). It was curious that satisfactory running could be obtained under those conditions in view of the effects of finish shown by Mr. Evans.

With reference to the paper by Messrs. McNicol, Williams, and Lamarque, Mr. Clayton said it was unfortunate that any suggestions regarding the tests involved complication, as the present method was so delightfully simple. The tests described by Moutte, Dixmier, and Lion ‡ indicated, however, the importance of low- as well as high-temperature treatment in affecting the type of oxidation product. Also immediate

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* V.D.I. Forschungsheft No. 391, 1938.

† *Jl. App. Mech.—Trans. A.S.M.E.*, **2**, 1935, p. A49.

‡ *I. Mech. E.*, "General Discussion on Lubrication," 1937, vol. 2, p. 372.

high-temperature treatment prevented the volatile portion playing any part in the oxidation process.

The agreement with the service order of six lubricants as regards chemical stability, and the correspondence Mr. Wilford (p. 610) found between hard asphalt in service and in the Air Ministry oxidation test, indicated that correlation ought to be possible considering the new test as an oxidation test. A number of other dish tests had been made, *e.g.*, by the Bureau of Standards and by van Hinte (mentioned by Mr. Bouman), and it would be interesting to know whether there was any correspondence in the results. One of the obvious difficulties was the lack of knowledge of the oil used in each case, and they had there yet another example of the benefits that would accrue from the availability to all experimenters of two or three common reference oils.

Dr. H. Wright Baker wrote that Keyser and Miller, when speaking of cylinder wall temperatures had mentioned " another useful tool." He would go much further, and suggested that wall-temperature measurements, which could be made very simply, were essential to an understanding of many problems. The temperature of the coolant was almost useless as a guide to the temperature conditions or variations at the walls. A knowledge of piston temperatures, although much more difficult to obtain, was highly desirable, even if only to prevent the easy acceptance of theories which at first sight seemed obvious, but which were, in fact, frequently false.

The authors were to be congratulated on their contribution to the development of a simple and robust method of measurement which could be applied through long-period runs and which was largely free from the variables introduced by linkages, etc., in the piston mouth. Certain modifications might be suggested—for instance, the use of aluminium-constantan couples with aluminium pistons, coupled with a differential method of temperature measurement which would enable six contacts to serve for five couples instead of three.

It was to be regretted that although the method had been known for some years, the data regarding the apparatus were still so scanty—the drawings were published without any scale attached, and no mention was made of plunger mass and spring strength, which would probably be very relevant matters at really high speeds.

The same lack of data was very noticeable throughout the paper—for instance, Fig. 8 was published without reference to piston metal, and again without a scale, although the temperatures might vary with different pistons under the same test conditions and in the same engine from those which gave a very light varnish under the crown to others at which no carbonaceous deposit whatever could exist. He (Dr. Baker) could only recommend great caution in accepting the figures given in the paper until such time as the data necessary for their understanding were forthcoming.

A piston design was a compromise, but it should be a balanced compromise. Some of the worst cases of " lubrication " troubles would go when it was realized that one bright idea did not make a piston.

He hoped that the authors would continue their work of collecting and sifting facts, and would publish them with full details. There would be far less talk of " road tests " when they had taken the trouble to trace the variables which affected their problems. Engine tests were sufficiently complex without the introduction of chassis, road, weather, and personal factors. The importance of the temperature factor was very great.

Mr. J. H. Evans, in reply to the Discussion of his paper, wrote :

" As Mr. E. A. Evans points out in his summary of the papers, the term ' dope ' is very loosely used to apply to extreme-pressure additives in both oils and greases, and it is preferable that such agents should be referred to as E.P. addition agents or E.P. bases.

Although having a preference for non-separating lime-soap greases for the general lubrication of Timken bearings, it was not intended to convey that all and sundry lime-soap greases were satisfactory and soda-soap greases unsatisfactory. Conditions of application must always be carefully considered and recommendations adjusted accordingly. As Evans stated, a rigid specification has been drawn up, but it is significant, from the long list of approved manufacturers, that the grease-makers can consistently meet the requirements of such specifications.

It is gratifying to learn that the Timken Stability Test has become both popular and important, as this militates against the lone struggle that took place for such a long time.

In reply to Mr. D. Clayton, it is appreciated that variations in the surface finish may account for some of the differences between different E.P. testing machines. The question of surface finish of test components is very involved; experiments made on components which had been measured on the "Contoragraph" did not result in accordance with the rating of this instrument.

The effect of surface polish, as Clayton suggests, is to re-group the O.K. loads and coefficients of friction in a much higher plane than the ground components.

This helps to obtain uniformity of test components finish, but removes the comparison between testing machine and ordinary workshop finish of parts, unless 'Superfinish' becomes general practice for all ground or machined components.

The scars on the test blocks are shown in Plate I at their O.K. load.

Standard grind, fairly deeply worn scratched surface; I.F. similar appearance smaller area; O.D. and rouge small scars slight separated scratches.

With the exception of the standard grind, which showed the usual abrasive wear, the others consist of fine abrasive lines having very little depth."

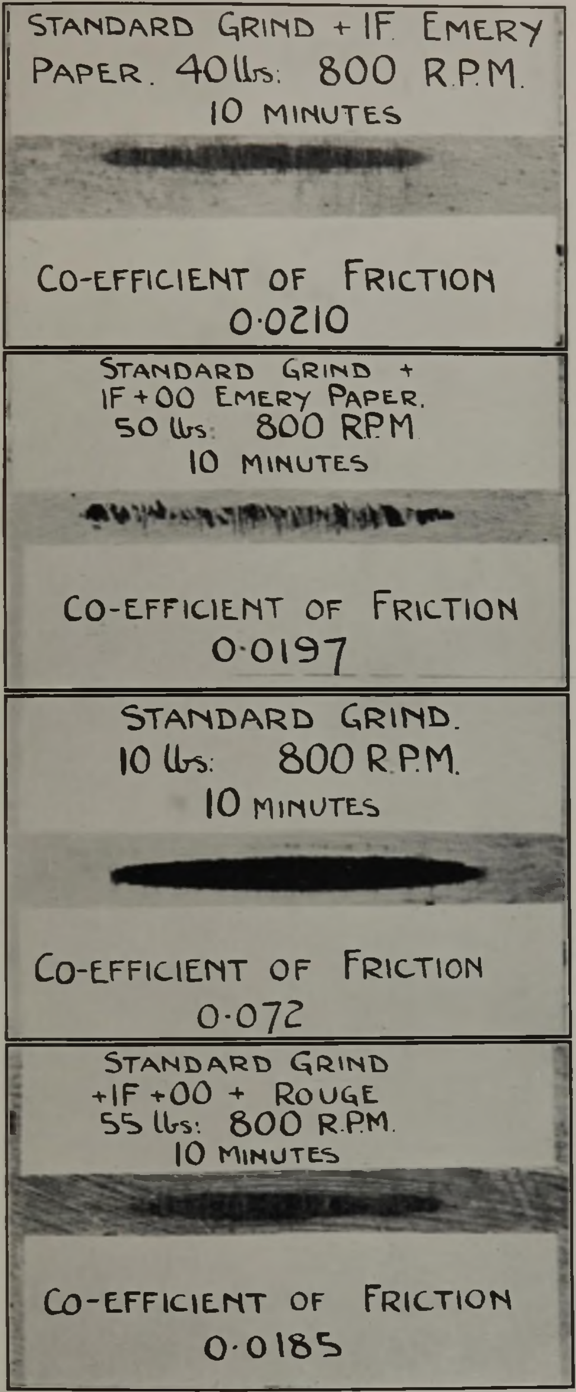

PLATE I.

[To face p. 800.

PREPARATION OF SOME PURE HYDROCARBONS FOR THE PURPOSE OF TESTING THE PHYSICAL METHODS IN USE FOR THE EXAMINATION OF HYDROCARBON MIXTURES.* (PART I.)

By H. I. Waterman, Hon.M.Inst.Pet., J. J. Leendertse, A.M.Inst.Pet., and D. W. van Krevelen.

Introduction.

In the first paper on this subject,† attention was directed to the applicability, within narrow limits,[1] of the specific refraction molecular-weight method [2] to the determination of the elementary composition of saturated hydrocarbon mixtures. Proof for this applicability was furnished by comparing the results of a great number of elementary analyses of highly divergent mixtures of hydrocarbons of high molecular weight, with the results obtained from the application of the specific refraction method. Naturally, the limits of accuracy between which this check could be effected depend on the limit of accuracy of the elementary analysis. The latter, however, is not so great as the accuracy that can be reached in the determination of the specific refraction, so that for a complete test of the specific refraction method it is necessary to carry out a more exact check in some other way. This can only be achieved by preparing a number of pure hydrocarbons of different types, and testing the specific refraction method on these hydrocarbons and on mixtures of them.

Moreover, testing the specific parachor molecular-weight method [3] also requires a knowledge of the properties of a number of pure hydrocarbons of different types. It is known that, according to the latter method, an indication would be found in regard to the degree of branching of hydrocarbon mixtures, by comparing the cyclic character of the mixtures derived by means of the specific refraction method with the cyclic character obtained by the application of a specific parachor molecular-weight diagram evolved for this purpose. As a matter of fact, branched products show an excess of rings in the specific parachor diagram. The magnitude of this excess is a measure of the branching.

In view of the above, the authors intend to prepare a number of pure hydrocarbons of different types, of which those discussed in this paper are the first.

A description is given below of the preparation of pure normal octadecyl benzene, from which, by hydrogenation, pure octadecyl *cyclo*hexane could be obtained. It is on the latter substance that the above specific refraction and specific parachor molecular-weight methods were applied. The results obtained were approximately the same as those calculated for the pure substance concerned. Thus the hydrogen content derived from the observed specific refraction (0·3304) amounted to 14·49 per cent.,

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* Paper received January, 1939.

† Compare "The Purity of some High Molecular Hydrocarbons as described in the Literature," by H. I. Waterman and J. J. Leendertse, *J. Instn Petr. Tech.*, 1939, **25**, 89.

whilst on the strength of the constitution a hydrogen content of 14·38 per cent. was to be expected.

The octadecyl *cyclo*hexane, however, differed considerably from the product prepared by Mikeska.[5] As a by-product in the preparation of the octadecyl benzene, hexatriacontane was obtained. This latter product, too, was analysed after further purification.

Moreover, the specific refraction and specific parachor molecular-weight methods were applied to a synthetically prepared, not absolutely pure octyl *cyclo*hexane, and also to the hexadecane obtained as a by-product in the preparation of the former. For all these hydrocarbons the methods gave the results expected.

DESCRIPTION OF THE INVESTIGATION.

As base materials for the synthesis there were available normal primary octyl chloride, normal primary octadecyl chloride and bromobenzene, which were thoroughly purified. The original idea was to prepare the above aromatics, which, when carefully hydrogenated, could supply the desired saturated naphthenes, by making the Grignard compound of the chlorides or bromobenzene, and reacting this with bromobenzene or the chlorides, respectively. It was found possible, in fact, to make the Grignard compound concerned, but condensation with the corresponding component met with considerable difficulty, so that another method was adopted for the preparation. As such the Würtz–Fittig reaction was selected, in which, starting from a mixture of bromobenzene with the chlorides, the aromatic in question was formed. For preparing octyl benzene this reaction was carried out in the presence of ether; for preparing octadecyl benzene this dilution with ether could be omitted.

In addition to the reaction product desired, diphenyl (resulting from the reaction of 2 mols. of bromobenzene) and hexadecane and hexatriacontane, respectively (resulting from the combination of two chloride molecules), were also obtained in the reactions. Owing to these additional reactions the octyl benzene obtained could not be separated in an entirely pure state, as the impurities were very difficult to separate from the required product, due to the slight differences in the boiling points. The hexadecane preparation obtained as a by-product is to be considered as a *practically* pure product. Therefore, even if these preparations had no direct value for testing the specific refraction method, they were quite suitable for checking the specific parachor method, in so far as it consists in comparing the cyclic character of the products derived from the specific refraction and specific parachor molecular-weight graphs.

More favourable were the results obtained in the preparation of the octadecyl benzene and the hexatriacontane formed as a by-product in this preparation. In this case the additional reaction resulting in the formation of the paraffin was a very welcome one, as it ensured simultaneous, rapid, and reliable preparation of the hexatriacontane.

Besides the above additional reactions, which were expected, there was another reaction, although occurring to a slight extent only, which will be discussed later in greater detail.

BASE MATERIALS.

The bromobenzene used was a commercial preparation, purified by repeated subjection to sharp fractionation at atmospheric pressure. A preparation was finally obtained with a boiling point (760 mm.) of 155·2–155·9° C., $n_D^{20} = 1{\cdot}5599$, $d_4^{20} = 1{\cdot}4914$, molecular refraction 34·03 (calc. 34·07), and this was taken as a base material for the experiments.

As normal primary octyl chloride a preparation made by Fränkel and Landau was used (quality " purest "). Although its appearance was fairly pure, this product was also subjected to a repeated fractionation at atmospheric pressure. The final preparation used in the syntheses had the following constants: boiling point (755 mm.) 182·8–183·6° C., $n_D^{20} = 1{\cdot}4306$, $d_4^{20} = 0{\cdot}8704$, molecular refraction 44·15 (calc. 44·01), chlorine content 24·1 per cent. (calc. 23·9%).

The purity of this substance was further evidenced by the fact that its melting point was entirely constant and coincided with its solidifying point.

The normal primary octadecyl chloride, too, was a commercial preparation made by Fränkel and Landau, and it was also purified. In this case purification by distillation was not really practicable, as even when distilling under a considerably reduced pressure some decomposition was found to take place. Purification of this substance could, however, be effected by fractional crystallization. Finally, a preparation was obtained with a constant solidifying point, or melting point, respectively, of 19·5° C. The preparation had n_D^{39} 1·4450, n_D^{40} 1·4445, n_D^{41} 1·4440, d_4^{41} 0·8490, specific refraction (41°) 0·3129, (20° corrected) 0·3124 (calc. 0·3123).*

PREPARATION OF OCTYL BENZENE, OCTYL *cyclo*HEXANE AND HEXADECANE.

34·5 gm. sodium wire were introduced into 250 cm.³ absolute ether, and, after cooling in ice-water, a mixture of 63 cm.³ bromobenzene and 124 cm.³ octyl chloride were added. The flask was then fitted with a Liebig cooler. After some time the sodium began to darken and heat was generated to such an extent that the ether began to boil and even partial distillation took place. When the reaction had proceeded for about 18 hrs., the excess sodium was decomposed with alcohol, the salts formed were dissolved in water, the ether layer was separated off, the water layer extracted with fresh ether, and the ether layer combined with the ether extracts dried on anhydrous Na_2SO_4. This complete treatment was repeated twice in exactly the same manner. After evaporation of the added ether, the total reaction product was subjected to distillation at 16 mm. In order to give an idea of the proportions in which the products had formed, the result of this first distillation is given below :

boiling point	40–70° (16 mm.)	n_D^{20}	1·4260	about	80 cm.³
,, ,,	120–150° ,,	,,	1·4856	,,	130 ,,
,, ,,	150–160° ,,	,,	1·4650	,,	40 ,,
,, ,,	160–168° ,,	,,	1·4565	,,	20 ,,

From this result we assumed that complete separation of the components

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* A more exact proof for the structure of this base material will be given in a following paper.

by distillation would meet with serious difficulties. The fractions obtained were subjected to numerous redistillations, and finally 66 cm.[3] of an " octyl benzene " preparation were obtained, with a boiling point of 140–144° C. (16 mm.). In view of the large boiling range, this is by no means to be considered as an entirely pure substance. The properties of the preparation are given in the table. Since a more satisfactory method of purification was not available in this case, the above product was used in the preparation of the " octyl *cyclo*hexane."

For this purpose the product was subjected to careful hydrogenation under high hydrogen pressure (initial pressure about 150 kilos/cm.^{2}), in the presence of 10 per cent. nickel catalyst (nickel on kieselguhr), the temperature remaining below 200° C. for the greater part of the hydrogenation. Only when the product was almost completely saturated was the temperature raised for some time above 200° C. (maximum 255° C.). The constants of the product thus obtained were determined; they are also given in the table.

The higher-boiling fractions of the reaction product of the Würtz–Fittig reaction were expected to consist essentially of hexadecane. Actually in the above fractionations of the reaction mixture two fractions were separated, the properties of which approximated those of hexadecane as mentioned in the literature, but which possibly still contained some octyl benzene, an impurity which is very difficult to separate from it by distillation.

Besides this, however, another impurity had to be considered; when determining the bromine number of one of the " hexadecane fractions," it showed a certain degree of unsaturation, pointing to the presence of olefinic double bonds. By treating the preparations with concentrated sulphuric acid and subsequently separating them with 5 per cent. fuming sulphuric acid and subjecting them to sharp fractionation, the products shown in the table as hexadecane I and hexadecane II are finally obtained.

In view of the process adopted, the hexadecane II is to be considered as the purest hexadecane preparation made by us; it had a constant melting point and solidifying point coinciding with each other. The other properties tallied well with those of the pure normal hexadecane described by Van Westen. Therefore, at any rate, the conclusion is justified that the hexadecane II is to be considered as a practically pure normal hexadecane.

The discovery of slight unsaturation due to olefinic double bonds in one of the " hexadecane " preparations led us to consider the Würtz–Fittig reaction in greater detail.

In order to find out whether the formation of the unsaturated compounds was caused by a possible splitting off of hydrocloric acid from the octyl chloride, the latter was made to react with sodium using ether as a medium, in a manner comparable with the process adopted when octyl benzene was formed. In the distillation of the reaction product first runnings were indeed found to be present, containing, in addition to unconverted octyl chloride and octene, also octane. The " hexadecane " separated by distillation also proved to contain a small amount of olefine (bromine number, according to McIlhiney, 8·5). Therefore, in the reactions in which octyl chloride enters into contact with sodium under the conditions referred to above, it appears that we have to consider a reaction comparable with that in which hydrochloric acid is split off, octene being formed at the same time. This octene might then be hydrogenated to normal octane by the hydrogen formed from sodium and the acid, whilst another part of the octene might have formed the hexadecene by polymerization; the latter might be partly recovered directly in the form of hexadecane by hydrogenation. The possibility of a branched hexadecene being formed partly when the octene is polymerized as stated above must not be precluded. On the other hand, the agreement found between the hexadecane II and the preparation made by Van Westen (see table) in quite a different way, and renders the presence of branched hydrocarbons in the preparations used for checking purposes highly improbable. Moreover, if the reaction scheme drawn up by Schlubach and Goes [4] for analogous reactions described in the literature should also hold good for this case, the formation of branched hydrocarbons would be out of the question.

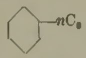
Finally it may be stated that high-pressure hydrogenation was carefully carried out on the hexadecene–hexadecane mixture obtained, with nickel as a catalyst, the highest temperature being 162° C. The product found has been called hexadecane III in the tables. Although this preparation was not purified with the same care as the previous hexadecane preparations, the specific refraction and the specific parachor prove to tally practically entirely with those of the other hexadecane preparations.

PREPARATION OF OCTADECYL BENZENE, OCTADECYL *cyclo*HEXANE AND HEXATRIACONTANE.

For the preparation of octadecyl benzene the base material consisted of 62·5 gm. sodium wire, 133·5 gm. bromobenzene and 350 gm. octadecyl chloride. Immediately after the sodium was introduced, the flask was placed in ice water and provided with a reflux cooler. In this case the reaction was slow; after the flask had been left to stand at low temperature for some 18 hours, in order to carry through the reaction as far as possible, the whole was heated up to 150° C. Thereupon the unconverted sodium was decomposed with alcohol, the separated salts dissolved in water, and the water layer extracted with fresh ether. In this treatment part of the reaction product remained undissolved; a large quantity of solid organic matter was left. This product was filtered off, 70 gm. of a substance with a melting point of 72° C. being separated.

The portion soluble in ether was distilled, first under atmospheric pressure to remove the ether, then at a pressure of 2 mm. First 8 gm. distillate were obtained, then 75 gm. unconverted octadecyl chloride (boiling point 175–190°, 2 mm.), and the rest distilled over almost completely at 212–216° C. The latter substance was the octadecyl benzene, of which some 70 gm. were obtained. This impure octadecyl benzene was purified by repeatedly subjecting it to crystallization. The best process that could be adopted for this purpose was dissolving the substance in ether and precipitating it again from the ether solution by means of methanol. This was repeated until the melting point remained entirely constant. The product thus obtained was a pure white powder. The constants are shown in the table, in which the data found by Mikeska are also mentioned.

This octadecyl benzene was used for the preparation of pure octadecyl *cyclo*hexane. To this end the substance was subjected to careful hydrogenation under high hydrogen pressure and with nickel catalyst, in the same way as mentioned for the preparation of the octyl *cyclo*hexane. The maximum temperature in this case was 260° C.*

Compound.	n^t_D.		$d^t/_4$.		r^t_D.		r^{20}_D. Theoretical.	Aniline Point. °C.		Molecular Weight.	
	n_D.	t° C.	d.	t° C.	r_D.	t° C.		Observed.	From Graph.	Observed.	Theoretical.
" *n*-Octyl benzene" 	1·4890	20	0·8622	20	0·3348	20	—	< − 5	—	—	190·18
" Octyl *cyclo*hexane "	1·4515 1·4311	20 70·3	0·8165 0·7822	20 70·3	0·3301 0·3310	20 70·3	0·3294 —	74·7		195	196·22
" Hexadecane " I	1·4354	20	0·7752	20	0·3369	20	0·3363	—	—	—	—
" Hexadecane " II	1·4351	20	0·7746	20	0·3369	20	0·3363	95·0	95	—	—
" Hexadecane " III	1·4345	20	0·7742	20	0·3367	20	0·3363	—	—	—	—
Hexadecane literature	1·4352 [a]	20	0·7751 [a]	20	0·3368 [a]	20	0·3363	—	—	—	—
Octadecyl benzene	1·4778	27									
	1·4768	30	0·8437	41	0·3322	41	—				
	1·4745	36	0·8224	71·5	0·3335	71·5		50·7	—	—	330·33
	1·4730	40			(extra-pol.) 0·3313	20					
	1·4640	63									
	1·4600	73·5									
Octadecyl benzene literature	1·4812 [c]	25	0·8540 [c]	25	0·3333 [c] (calc.)	25	—	47·0 [c]	—	—	—
Octadecyl *cyclo*hexane	1·4555	33·8	0·8126	48	0·3310	48		106·7	106	—	336·38
	1·4542	38									
	1·4522	43	0·7983	70·3	0·3315	70·3					
	1·4500	49									
	1·4420	70·3			0·3304 (extr.)	20	0·3294				
Octadecyl *cyclo*hexane literature	1·4538 [c]	25	0·8340 [c]	25	0·3246 [c] (calc.)	25	0·3294	—	—	—	—
					0·3245 (extr.)	20					
Hexatriacontane	1·4359	77									
	1·4349	80	0·7795	82	0·3341	82		132·8	126	—	506·59
	1·4341	82	0·7671	100	0·3348	100					
	1·4316	88			0·3319 [b] (extr.)	20	0·3325				
	1·4313	89									
Hexatriacontane literature	—	—	0·7819 [d]	76	—	—	—	—	—	—	—

[a] H. A. v. Westen, Thesis, Delft, 1931, p. 70. H. I. Waterman, P. van 't Spijker, and H. A. van Westen, *Rec. trav. chim.*, 1929, **48**, 1103.
[b] P. C. Carey and J. C. Smith, *J. chem. Soc.*, 1933, 346.
[c] L. A. Mikeska, *Ind. Eng. Chem.*, 1936, **28**, 970.
[d] F. Krafft, *Ber.*, 1907, **40**, 4779.
[e] Gascard, *Compt. rend.*, 1911, **153**, 1486.

The reaction product of the hydrogenation was recrystallized in the same way as described for the corresponding unsaturated product. The

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* It has since been proved that this hydrogenation at high temperature in the presence of nickel catalyst has not caused undesired side-reactions (compare H. I. Waterman, J. J. Leendertse, and J. F. Sirks " Preparation of some Pure Hydrocarbons, etc.", Part II, p. 809.

substance finally obtained consisted of small leaves with a silvery lustre, the properties of which are shown in the table, together with the constants mentioned by Mikeska.

Surface Tension[i] t° C.		Specific Parachor.		Specific Parachor from graph.	Specific Dispersion $\times 10^4$.	Number of rings per molecule.			Boiling Point, ° C.	Melting Point, ° C.	Halogen Content, %.	
σ(dyne/cm.).	t° C.	p.	t° C.			From r_D^{20}.	p^{20}.	Theoretical.				
30·59	20	2·728	20	—	239	—	—	—	140–144 (16 mm.)	—	—	The authors' preparations
29·17	20	2·846	20	2·846	154·5 (70·3)	0·9	1	1	—	—	0	
27·66	20	2·959	20	2·962	—	−0·1	0	0	160–162 (15 mm.)	17·0	—	The authors' preparations
27·67	20	2·961	20	2·962	—	−0·1	0	0	157–158 (15 mm.)	17·8	—	
27·50	20	2·958	20	2·962	—	−0·05	0	0	—	14·8	—	
—	—	—	—	—	—	−0·1	—	0	—	18·13[b]	—	
										17·8[a]		
30·85	50	2·815	50	—	—	—	—	—	—	32·8	—	The authors' preparation
28·88	71·5	2·819	71·5									
		2·809 (extr.)	20									
—	—	—	—	—	—	—	—	—	—	35–36[c]	—	
29·75	48	2·874	48		153	0·8	0·9	1	—	40·7–40·8	0	The authors' preparation
28·26	70·5	2·889	70·5									
		2·855 (extr.)	20	2·847								
—	—	—	—	—	—	2·0	—	1	—	40[c]	—	
27·48	84	2·943	84		—	(0·2)[h]	(0·2)[h]	0	—	76·0	0	The authors' preparation
26·59	98	2·955	98									
		2·888[h] (extr.)	20	2·901								
—	—	—	—	—	—	—	—	—	—	about 76·0[d]	—	
										76·0[e]		
										76·5[f]		
										78·5[g]		

[f] A. Oskerko, *Chem. Zentr.*, 1914, II, 1265.
[g] P. A. Levene, C. J. West, and J. v. d. Scheer, ref. in *Brit. Chem. Abstr.*, 1915, **34**, 634.
[h] Extrapolated over a wide temperature range, and therefore not very accurate.
[i] The determinations were made by the stalagmometric method.
[j] This preparation did not contain any sulphur .

Finally it was necessary to further purify the solid substance, only slightly soluble in ether, which was separated from the total reaction product and which, in view of the way in which it was formed, may be considered to be impure hexatriacontane. This product, coloured slightly brown, was recrystallized a few times from petroleum ether, after the solution had been treated with norite. Finally the melting point became constant at 76·0° C. The product consisted of pure white leaves and was fairly readily soluble in warm petroleum ether and warm benzene, but soluble with difficulty in ether and alcohol. The constants of this substance are also given in the table.

It may be remarked that in this synthesis no indications were found of the formation of olefinic unsaturated compounds.

Consideration of the Results Obtained.

It follows from the results obtained that for the compounds considered the number of rings per molecule may be derived from the specific refraction, with an accuracy of at least 0·2 ring per molecule.

For the calculation of the specific refraction of hexatriacontane at 20° C. it was necessary to extrapolate over a very large temperature range, therefore in this case the latter specific refraction value may be somewhat less accurate than for the octadecyl *cyclo*hexane. Yet the results obtained could still be qualified as highly satisfactory, as the specific refraction gave an indication of 14·66 per cent. hydrogen, whilst from the constitution 14·72 per cent. might be expected.

It is apparent from the specific parachor values found that the specific parachor tallies well with the specific parachor molecular-weight diagram previously drawn up. Here, too, the result for hexatriacontane is slightly less accurate than for the other compounds, owing to the large extrapolation required for calculating the specific parachor at 20° C.

When two compounds of a homologous series differing appreciably in molecular weight correspond in properties, it is not probable that the other compounds belonging to this series will show deviations, therefore we may consider this check as a check on the curve of the unbranched paraffins and on the curve of the monocyclic six-ring naphthenes with only one unbranched side-chain. For the compounds in question, when calculating the number of tertiary C atoms by a comparison of the number of rings per molecule derived from the specific refraction and also from the specific parachor, about 1 tertiary C atom would be found, which, within the limits of accuracy of observational error, agrees with the actual content.

References.

[1] H. I. Waterman and J. J. Leendertse, *Chimie et Industrie*, numéro spécial congrès Paris, September 1937.

[2] J. C. Vlugter, H. I. Waterman, and H. A. van Westen, *J. Instn Petr. Tech.*, 1935, **21**, 661.

[3] H. I. Waterman and J. J. Leendertse, *ibid.*, 1938, **24**, 16.

[4] H. H. Schlubach and E. C. Goes, *Ber.*, 1922, **55**, 2889.

[5] L. A. Mikeska, *Ind. Eng. Chem.*, 1936, **28**, 970.

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PREPARATION OF SOME PURE HYDROCARBONS FOR THE PURPOSE OF TESTING THE PHYSICAL METHODS IN USE FOR THE EXAMINATION OF HYDROCARBON MIXTURES.* (PART II.)

By H. I. WATERMAN, Hon.M.Inst.Pet., J. J. LEENDERTSE, A.M.Inst.Pet., and J. F. SIRKS.

SUMMARY.

The *n*-octadecyl*cyclo*hexane prepared in a pure state from *cyclo*hexyl iodide and octadecyl chloride has been shown to be identical with the *n*-octadecyl*cyclo*hexane prepared from *n*-octadecyl benzene by hydrogenation at 200–260° C., using high-pressure hydrogen and nickel-on-kieselguhr as a catalyst.[1] Thus, hydrogenation under these conditions did not cause undesirable structural changes. This fact is of general interest, because similar hydrogenations are often carried out in the analysis of hydrocarbon oils using the specific refraction–molecular weight method.[2]

INTRODUCTION.

THE synthesis of pure *n*-octadecyl*cyclo*hexane has been described in a previous paper.[1] For the preparation of this compound, the Wurtz–Fittig condensation of normal primary octadecyl chloride and bromobenzene was used, followed by purification of the octadecyl benzene. In order to obtain *n*-octadecyl*cyclo*hexane from the octadecyl benzene, the latter substance was hydrogenated at about 200–260° C., using high-pressure hydrogenation and nickel-on-kieselguhr as catalyst. After two recrystallizations of the hydrogen product (yield of these recrystallizations more than 90 per cent.), the resultant compound was considered to be pure *n*-octadecyl*cyclo*hexane.

However, during the course of hydrogenation, owing to the relatively high temperature and the presence of nickel catalyst, it is possible that isomerization or other structural changes might have taken place during the reaction. In order, therefore, to prove the reliability of the method of preparation of *n*-octadecyl*cyclo*hexane, previously described,[1] the authors decided to synthesize the same compound in another way, avoiding hydrogenation at high temperatures—namely, by the condensation of *cyclo*hexyl iodide and *n*-octadecyl chloride, using metallic sodium.

The condensation product agreed very well with the *n*-octadecyl*cyclo*hexane previously described,[1] and it was therefore concluded that the structure of *n*-octadecyl benzene had not been changed by hydrogenation. This fact is also of general interest, in view of the fact that similar hydrogenations are often carried out in the analysis of hydrocarbon oils using the specific refraction–molecular weight method.[2]

EXPERIMENTAL.

The method of preparation of *n*-octadecyl*cyclo*hexane described below is similar to the method recently [1] followed for the synthesis of pure

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* Paper received January 1939.

n-octadecyl benzene. *cyclo*Hexyl iodide was, however, used instead of bromobenzene. (*cyclo*Hexyl bromide could not be used, as the yields proved to be very poor.)

The base material " normal primary octadecyl chloride " was a commercial product made by Fränkel and Landau (quality " reinst "), similar to the preparation used in the experiments previously described.[1] It was recrystallized once. [After recrystallization : $n_D^{25} = 1{\cdot}4509$; $d_4^{25} = 0{\cdot}8579$; specific refraction (25° C.) $= 0{\cdot}3137$, (calculated for $C_{18}H_{37}Cl$) 0·3123; melting point = 19·4° C.]

The *cyclo*hexyl iodide was prepared by interaction of equimolar quantities of *cyclo*hexanol (Fränkel and Landau, quality " reinst "), and a 70 per cent. hydriodic acid solution at room temperature, followed by heating at 105° C. for $1\frac{1}{2}$ hour, the reaction mixture being stirred. The lower layer of the reaction product was washed with a potassium hydroxide and a sodium thiosulphate solution. After drying over anhydrous Na_2SO_4, it was distilled. A fraction with boiling point 33° C. (1 mm.) and n_D^{20} 1·548 was used in the experiment, although it was probably contaminated with some *cyclo*hexanol.

The condensation experiment was carried out in a 1-l. round flask with reflux cooler. 14·2 g. sodium (wire) were introduced in the (cooled) mixture of 52 g. *cyclo*hexyl iodide and 71·5 g. octadecyl chloride. The reaction temperature was kept at about 60° C. during the main part of the reaction. When the condensation was nearly finished the whole was heated at 130° C. for 1 hour.

After cooling, the unconverted sodium was decomposed with alcohol, the separated inorganic salts, the alcohol, etc., dissolved in water, and the water layer, as well as the hydrocarbon layer, extracted with cold ether. In this treatment a part (25·5 g.) of the product remained undissolved. Probably this part (melting point 75° C.) consisted mainly of hexatriacontane, obtained by the reaction of 2 mols. of n-octadecyl chloride (compare also [1]).

The solution of the hydrocarbons in ether was dried over anhydrous Na_2SO_4 and distilled first under atmospheric pressure to remove the ether, and then at a pressure of 1–2 mm. The result of this distillation is summarized in Table I.

TABLE I.

Fraction Number.	Quantity, g.	Boiling Temp., ° C.	Distillation Pressure, mm.	n_D.	Melting Point, ° C.	Remarks.
I	6·5	80–95	1–2	1·4757 (20°)	—	Di*cyclo*hexyl ?
II	16·5	144–149	,,	1·4368 (35·2°)	24·7	Probably mainly octadecane and octadecene
III	1	149–184	,,	—	—	—
IV	20·5	184–190	,,	1·4538 (40°)	38·8	Octadecyl*cyclo*hexane
Residue	5	> 190	,,	—	—	—

The melting point and n_D values of the fourth fraction are practically the same as those for pure octadecyl*cyclo*hexane (compare [1]). This fraction was purified by several recrystallizations, the first time by dissolving it in ether and precipitating it from the ether solution by means of methanol,

TABLE II.

Octadecylcyclohexane.

Product from	n^t_D.		d^t_4.		Specific Refraction.		Surface Tension.		Specific Parachor.		Solidifying Point, ° C.	Specific Dispersion.*	Number of Rings per Molecule.		
	n_D.	t° C.	d.	t° C.	r^t_D.	t° C.	σ_t (dyne/cm.).	t° C.	p_t.	t° C.			from r^{20}_D.	from p_{20}.	Theoretical Value.
Octadecylbenzene, prepared by D. W. van Krevelen.[1]	1·4504 1·4420	48 70·3	0·8126 0·7983	48 70·3	0·3310 0·3315 0·3304	48 70·3 20	29·75 28·26	48 70·5	2·874 2·889 2·855 (extrapol.)	48 70·5 20	40·7–40·8	153	0·8	0·9	1·0
Octadecylchloride and *cyclo*hexyl iodide prepared by J. F. Sirks.	1·4503 1·4423	48 70	0·8124 0·7983	48 70	0·3310 0·3316 0·3302	48 70 20	29·80	48	2·876 2·857 (extrapol.)	48 20	40·9	152	0·8	0·9	1·0

$$* \quad \frac{n_{g'} - n_C}{d} \times 10^4.$$

followed by simple recrystallizations from ether as such. These recrystallization experiments were continued until the physical constants remained practically constant, which was the case after four recrystallizations.

In Table II the physical constants of the final product are compared with those previously described.[1] The figures in this table clearly indicate that the two preparations prepared by two investigators using different methods of synthesis are identical.* It can therefore be concluded that the hydrogenation of pure *n*-octadecylbenzene at 200–260° C. using high-pressure hydrogen and nickel-on-kieselguhr as catalyst has not changed the original structure of this hydrocarbon. Moreover, the agreement between the physical constants of both products is valuable evidence of the accuracy of the constants for pure *n*-octadecyl benzene and *n*-octadecyl*cyclo*hexane, previously described.[1]

References.

[1] H. I. Waterman, J. J. Leendertse, and D. W. van Krevelen, *J. Instn Petrol.*, 1939, **25**, 805.

[2] J. C. Vlugter, H. I. Waterman, and H. A. Van Westen, *J. Instn Petrol. Tech.*, 1935, **21**, 661.

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* It may also be noted that the melting point of a mixture of equal quantities of the preparations of Sirks and van Krevelen[1] agreed completely with the melting points of these individual samples.

CORRESPONDENCE.

THE ANALYSIS OF THE KEROGEN OF OIL SHALES.*

Mr. F. W. Quass (*Fuel Research Institute of South Africa*) writes :

Incidental to some work being undertaken at the Fuel Research Institute of South Africa, it has been necessary to attempt the reduction of the mineral matter of certain coals and oil shales (torbanites). The nature and the object of the research made it impossible to use acid extraction as carried out by Dr. Down. By applying a method based on the Trent Amalgamation Process, success has been achieved in removing the greater portion of the mineral matter from these samples.

The method is briefly as follows : A sample of the torbanite is very finely ground with water in a porcelain ball mill and then transferred to a porcelain end-runner, where a quantity of oil is added. All the torbanite, after grinding, passes through 200 mesh, and the average particle size is very much smaller. At this stage the mass consists of 50 gms. of torbanite, 200 c.c. of water and 60–70 c.c. of oil. The oil forms a paste with the carbonaceous material, and the greater portion of the mineral matter remains suspended in the water and can be separated. The separation of low-ash carbonaceous material from the mineral matter is complete, the mineral matter showing no trace of dark colour. The oil-paste is thoroughly cleaned by washing with water while agitating it by means of an egg-beater. The oil is then removed by washing the paste with hot petrol and finally with carbon tetrachloride. The first separation is extremely efficient, reducing the ash content of South African torbanite from 40 per cent. to about 10 per cent. The process, however, may be repeated successfully, and eventually a minimum ash content is obtained. This minimum figure is reproducible, and may be provisionally regarded as representing the " organic " or " inherent " mineral matter content of the torbanite.

Analyses of two samples of torbanite before and after treatment are given below in Tables A and B.

Table A.

Proximate Analysis.

	Sample 1.		Sample 2.	
	Whole.	De-ashed.	Whole.	De-ashed.
% Moisture	0·9	4·2	1·2	1·7
% Ash	31·2	9·7	37·6	4·9
% Volatile Matter	49·9	61·0	42·7	62·5
% Fixed Carbon	18·0	25·1	18·5	30·9
% V.M. (Dry ash-free)	73·5	70·8	69·8	66·9

In Table A it is noticed that the percentage of volatile matter calculated on the dry, ash-free torbanite decreases on the treated samples.

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* Paper by A. L. Down, D.Sc., in the April 1939 issue of the Journal (pp. 230–237).

TABLE B.
Ultimate Analysis.

	SAMPLE 1.		SAMPLE 2.	
	Whole.	De-ashed.	Whole.	De-ashed.
% Carbon D.A.F.	80·3	85·9	79·2	83·0
% Hydrogen D.A.F.	9·16	9·00	8·52	8·27
% Oxygen D.A.F.	8·2	3·1	8·2	6·1
C/H ratio	8·8	9·5	9·3	10·0
C/O ratio	9·8	27·7	9·7	13·6

The ultimate analyses indicate that similar conclusions to those reached by Dr. Down can be made, namely :—

(1) the percentage of carbon is higher in the treated samples,
(2) the percentages of hydrogen and oxygen are lower in the de-ashed samples, and
(3) the ratios of carbon to hydrogen and carbon to oxygen have increased in these de-ashed torbanite samples.

At present the solution offered by Dr. Down—viz., that the unknown amount of water of hydration of the mineral matter associated with the torbanite makes the ultimate analysis, as determined on the original samples, contain errors of considerable magnitude—seems the most feasible explanation of the anomalous results given in Tables A and B. From Table A it will be noted that the volatile matter content of the de-ashed torbanite is less than that for the original, and it seems a pity that Dr. Down did not carry out volatile matter determinations on the de-ashed samples as well as on his air-dried samples. This decrease follows also quite logically from the solution given above.

If, however, the liquids used to wash the oil out of the paste dissolve part of the organic material of the torbanite, the effect on the ultimate analysis of the samples would be a tendency to give higher carbon and oxygen and lower hydrogen contents, since the material extracted is likely to be rich in hydrocarbons of a high hydrogen content. For similar reasons the volatile matter content should decrease.

No study of this possibility with South African torbanites has as yet been made, and it must be recognized that the above argument cannot explain the remarkable decrease in the oxygen content of the de-ashed samples unless very large quantities indeed of extract are removed by the solvents.

Altogether, at this stage it is felt that the order of any effects due to solution is insufficient to account for the differences which obtain, and preference is given to the explanation advanced by Dr. Down.

Dr. G. W. HIMUS (*Elveden Fuel Laboratories, Imperial College, London*) writes :—

MR. Quass is to be congratulated on having worked out a method of reducing the mineral content of oil-shale by a physical method which can be conveniently applied to considerable quantities of material, while at the same time it should be possible to make a substantially quantitative separation of the kerogen from the mineral matter, the latter being recovered in such a condition that it can be examined in detail. The process of removing the mineral matter by means of treatments with acids (A. L. Down, *Journal Inst. Petroleum,* 1939, **25,** 230–237) suffers from the inherent disadvantages that it is costly and difficult to apply to more than 30 to 50 gms. of sample at one time, and also that during the treatment with hydrofluoric acid to remove silica, the silica is not easily recoverable, ; consequently, it is generally not feasible to carry out a weight-balance throughout the de-ashing.

It would be a matter of interest if Mr. Quass would give quantitative data (*a*) as to the percentages of kerogen and mineral matter recovered from his specimens and also (*b*) as to the nature of the mineral matter separated. The statement that the mineral matter showed no traces of dark colour is insufficient and might well be amplified.

While, however, the practical value of Mr. Quass' work is not in question, there are one or two statements in his communication which are open to criticism. Mr. Quass speaks of " the anomalous results given in Tables A and B." The " anomalous results " presumably are the differences in the compositions of the dry, ashless materials as calculated from the analyses of the " whole " and the de-ashed samples, and he directs attention more particularly to higher carbon and the " remarkable decrease in the oxygen content of the de-ashed samples."

In point of fact there is no anomaly in the results ; the calculation of the composition of a solid fuel on a " dry, ash-free " basis is really an attempt to arrive at the true composition of the organic matter in the fuel—*i.e.*, free from moisture and *mineral matter.* But, inasmuch as the ash as determined by incineration is invariably less in quantity and different in composition from the original mineral matter, the composition of the organic component of the fuel as calculated " ash-free " will always differ from the true composition, the difference being a function of the mineral content of the sample on which the analysis was carried out. The errors introduced by this difference between the percentages of ash as determined by incineration and original mineral matter, *plus* small errors inherent in any analysis, are summed in the " oxygen " figure which is arrived at by deducting from 100 the total of the carbon, hydrogen, nitrogen, sulphur, and ash. " Oxygen " should be reported as " oxygen by difference " or (better) " oxygen *plus* errors," and, as Mr. Quass' results show, the " oxygen *plus* errors " may consist chiefly of errors with a little oxygen thrown in as a makeweight. If, however, prior to carrying out an analysis, the fuel is treated so as to reduce the ash to (say) 2 per cent. or less, the errors are reduced, so that the final composition as calculated is a very close approximation to that of the pure organic matter.

Mr. Quass also remarks that Dr. Down's suggestion " that the unknown amount of water of hydration of the mineral matter associated with the torbanite makes the ultimate analysis, as determined on the original samples, contain errors of considerable magnitude—seems the most feasible explanation of the anomalous results given in Tables A and B."

At the outset of the research at the Imperial College, it was realized that until the uncertainties resulting from the presence of unknown quantities of hydrated minerals containing unknown amounts of combined water could be eliminated, it was hopeless to expect to be able to determine either the percentage of kerogen in a shale or its composition. The de-ashing process described by Dr. Down was therefore devised, and is now applied (with suitable modification, where necessary) to all samples of oil-shale which are to be investigated.

Mr. Quass very properly refers to the possibility that some of the kerogen may be removed in solution by the oil during the de-ashing treatment. While this danger is a real one, it should be possible to ascertain by experiment whether with any particular sample such an error is likely to be introduced, and then to guard against it by suitable modification of the treatment.

The above remarks are not offered in any spirit of carping criticism; Mr. Quass has worked out a method which will place in the hands of research workers a valuable weapon whereby they may overcome the inherent difficulty arising from the association in oil-shales of (often) a relatively small quantity of kerogen with a large proportion of mineral matter. By the application of this " amalgamation " process it should be possible to separate the kerogen from the mineral matter in an oil-shale, so that each constituent may be thoroughly studied both qualitatively and quantitatively.

Dr. A. L. DOWN (*Department of Chemical Technology, Imperial College, London*) writes :—

The success of a physical method for separating the kerogen from the mineral matter of an oil shale or torbanite is of considerable importance, since, as compared with processes involving treatment with mineral acids, it has the advantages that larger amounts of material can be treated and the possibility of any chemical alteration of the organic matter being effected by the reagents is eliminated. A. E. Flynn [1] employed a flotation method for isolating " kerogen globules "—*i.e.*, algal colonies—from " stellarite," a Nova Scotia torbanite, but the product contained 13·85 per cent. mineral matter, and the results showed that there had been a differential separation of the organic material, the gangue having about 25 per cent. of organic matter, of much lower " volatile matter " to " fixed carbon " ratio, associated with it. In the preliminary investigation of de-ashing at the Imperial College the " float-and-sink " test for the washability of coals was applied to a sample of South African torbanite, ground to pass a 90-mesh sieve, and practically no separation was obtained, the lightest fraction having approximately 30 per cent. of ash and the heaviest 50 per cent. The success achieved at Pretoria, therefore, shows the importance of very fine grinding if physical means of separation are to be used; further, it indicates the fineness of the original material and the intimate mixing of the organic and inorganic constituents.

The method employed by Mr. Quass has the disadvantage that the part of the kerogen soluble in organic solvents is removed; extraction of South African torbanite with acetone yielded 1·6 per cent. by weight of the kerogen and with chloroform 0·9 per cent. The soluble material is considered to be mainly hydrocarbons; J. W. McKinney [2] examined the acetone extract from a New Brunswick oil shale and found it to contain 85 per cent. of hydrocarbons. As A. J. Carlson [3] has shown, there is the possibility that part of the kerogen is combined in some way, either physical or chemical, with the mineral matter. Solvent extraction of substantially de-ashed samples of Kimeridge and Middle Dunnet oil shales showed that the amounts of soluble material, calculated as percentage kerogen, were higher than those obtained from the untreated shales.

	Acetone Extraction.	
	Oil Shale.	De-ashed Sample.
Kimeridge	4·22%	6·45%
Middle Dunnet	5·69%	8·97%

The extracts are given as percentage of kerogen.

The results suggested that either part of the organic matter was physically combined with the mineral constituents, or that the acid treatment had had some chemical effect on the kerogen. To investigate the possibility of physical absorption, a known weight of Scottish shale oil, dissolved in acetone, was mixed into a paste with approximately ten times its weight of Stourbridge " mild " clay and the solvent allowed to evaporate slowly. After standing for one week, the mixture, which was hard and dry, was soxhlet-extracted with acetone; the solvent dissolved only 87 per cent. of the oil, the remainder being retained by the clay. The procedure was repeated using a sample of Stourbridge " strong " clay, with similar results, the percentage of oil retained being slightly higher. Thus the increase in the proportion of kerogen extracted from the de-ashed samples is probably due to part of the " soluble " material in the oil shales being physically absorbed on the mineral matter.

In order to determine whether the acid treatment of oil shales gave reproducible results, the process was carried out in duplicate on a sample of Amherst oil shale. The analyses of the two de-ashed samples, which are recorded in Table C, showed that, although the removal of the silicates had been accomplished to different extents, the compositions of the kerogens calculated from these results were substantially the same. Therefore it is unlikely that the reagents had had any chemical effect on the kerogen, as it is improbable that the organic matter would be attacked to the same degree in different experiments.

The acid treatment for de-ashing has been applied to a sample of South African torbanite, from Mooifontein 287 in the Ermelo district, and one of Kohat oil shale from the N.W.F.P. of India. In the case of these samples and the Amherst oil shale, the iron pyrites was removed by reduction with nascent hydrogen formed *in situ* by the addition of concentrated hydrochloric acid to an intimate mixture of the sample with zinc dust. The hydrogen sulphide evolved was absorbed in an ammoniacal cadmium chloride solution and the sulphur estimated volumetrically by the iodine and sodium thiosulphate method. Finally, the residue was thoroughly washed with dilute hydrochloric acid and a large volume of water, to remove the iron and zinc chlorides, dried and weighed. The analyses of these samples are given in Table D.

The small amounts of ash obtained by the combustion of the de-ashed South African and Kohat kerogens were bright red in colour, soluble in hydrochloric acid, and were practically pure ferric oxide. Treatment with zinc and hydrochloric acid and with nitric acid, sp. gr. 1·12, both failed to dissolve the remaining iron, and since G. A. Cave (unpublished research) has shown that both the methods remove iron pyrites quantitatively, the iron cannot be present as sulphide. As all attempts to extract the iron failed and the repeated treatments had apparently no effect on the percentage of ferric oxide obtained on combustion, its presence is strongly presumptive evidence that the iron is directly combined with the organic matter and is, therefore, a constituent of the kerogens of South African torbanite and Kohat oil shale.

Table C.

Analysis of Amherst Oil Shale, Burma.

	Untreated Sample.		De-ashed Sample 1.		De-ashed Sample 2.	
	A, %.	B, %.	A, %.	B, %.	A, %.	B, %.
Carbon	40·95	73·1	73·35	77·47	74·76	77·46
Hydrogen	6·14	11·0	10·46	10·11	10·77	11·16
Nitrogen	0·55	1·0	1·23	1·31	1·21	1·25
Sulphur	0·82	1·4	0·75	0·79	0·79	0·81
" Oxygen "	7·6	13·5	8·89	9·39	8·97	9·32
Mineral Matter	43·95	—	5·32	—	3·52	—
C/H ratio		6·6		7·0		7·0
C/O ratio		5·6		8·3		8·3

A = Composition as obtained by analysis.
B = Composition of kerogen calculated from A.

Examination of the compositions of the kerogens of the three samples of South African torbanite, two calculated from the analyses of the de-ashed samples prepared by Mr. Quass and the one given in Table D, show that they vary between very wide limits, particularly the oxygen values. On the other hand the compositions of the kerogens from four different seams of Scottish oil shales, recorded in Table VI page 236, are all very similar,

Table D.

Analysis of South African Torbanite and Kohat Oil Shale.

	South African.			Kohat.		
	A, %.	B, %.	C, %.	A, %.	B, %.	C, %.
Carbon	42·76	78·72	79·75	8·60	71·32	72·58
Hydrogen	4·55	7·72	7·82	1·34	7·96	8·11
Nitrogen	0·91	1·54	1·56	0·18	1·31	1·34
Sulphur	0·32	1·20	1·22	0·55	0·80	0·81
" Oxygen "	6·4	9·52	9·65	4·0	16·86	17·16
Mineral Matter	44·9	1·86	—	85·3	2·56	—
C/H ratio	9·4		10·2	6·4		9·0
C/O ratio	6·7		8·3	2·1		4·2

A = Analysis of original dry sample.
B = Analysis of de-ashed sample.
C = Composition of kerogen calculated from B.
Mineral matter = Ash on incineration plus carbon dioxide from carbonates.

and recent results from the de-ashing of three samples of Lower Dunnet oil shale, from Westwood, are in close agreement with those already reported. The differences in the analyses of the torbanite may be due to variations in the proportion of one particular source-material, which had a chemical composition markedly different from the average. Microscopic examination has revealed that in the organic matter of Scottish oil shales plant remains predominate, there being very few spore cases and algae, whereas in South African torbanite the vegetable debris is associated with large numbers of algal colonies. It is possible that differences in the ratio of algal bodies to general plant debris are responsible for the variations in the results obtained for the composition of the torbanite kerogen.

The very marked differences in the analyses of the kerogens as calculated from the results of determinations on untreated oil shales and the de-ashed samples are undoubtedly due to the effects of the water of hydration of the clays. It was realised from the first that this water was responsible for large errors in the composition of the organic matter and it was with the object of eliminating these errors that the isolation of the kerogen was attempted. In the de-ashing process employed at the Imperial College, the filtrates from the acid treatments were analysed and weight balances calculated, and no part of the kerogen could have been removed. Further, as is illustrated by Table E, the determination of carbon and hydrogen at each stage showed that the C/H ratio remained constant until the silicates were attacked.

TABLE E.

Carbon and Hydrogen on Middle Dunnet Oil Shale.

	Carbon, %.	Hydrogen, %.	C/H Ratio.
Dry sample . . .	12·98*	2·04	6·38
HCl treated . . .	15·08	2·37	6·35
HNO_3 treated . .	15·33	2·39	6·41
HF treated . . .	78·63	8·17	9·62

* Less carbon present as carbonate.

The additional results relating to the acid treatment for removing substantially all the mineral matter from oil shales indicate that the method is satisfactory. In the study of coals certain important relationships were developed from considerations of the ultimate analyses of a great many samples, *i.e.*, Ralston's[4] and Hickling's[5] diagrams, which showed the general continuity of the "peat ⟶ anthracite" series, and when the true compositions of a large number of oil shale kerogens have been obtained it may be possible to trace some parallel relationship for these materials.

References.

[1] A. E. Flynn, National Research Council, Canada, Rpt. 18, 1926.
[2] J. W. Mckinney, *J. Amer. chem. Soc.*, 1924, **46**, 968–979.
[3] A. J. Carlson, Univ. of California, Pub. in Eng., vol. 3, 1937.
[4] O. C. Ralston, U.S. Bureau of Mines, Tech. Paper No. 93, 1915.
[5] G. Hickling, *Trans. Inst. Min. Eng*, 1926, **72**, 261.

ABSTRACTS.

AUTHOR INDEX.

The numbers refer to the Abstract Number.

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

Geology.

1246.* Structural and Magmatic Processes in the Isostatic Layer. M. G. Hoffman. *Bull. Amer.Ass. Petrol. Geol.*,1939, **23,** 1320–1351.—The Isostatic Layer is here referred to the outer 60 miles of the earth. This is made up, from the surface, as follows: a layer of approximately 10 miles composed largely of sediments and granitic rock, followed by an intermediate layer, from 15 to 20 miles thick, of basic crystalline rocks (gabbros, etc.), and a 30-mile layer of glassy basalt or tachylyte. This tachylyte layer is thought to be universal, and extends from the base of the intermediate division to the top of a dunite layer, 1800 miles thick, which rests on the liquid core.

Folding and faulting of strata in the earth's crust—that portion above the isostatic layer—indicate that the crust has been shortened. This is shown by its collapse on a shrinking interior. The temperature of the isostatic layer remains essentially constant while the interior cools and shrinks. Stresses in the crust continue until breaking point is reached, when relief is brought by folding and faulting.

Mountainous areas are reduced by erosion, and down-warped areas are filled with great masses of sediments, isostatic balance being maintained during these processes.

The filling of basins and the reduction of mountain systems bring about changes in base level and the position of the isogeotherms. The amount of deepening caused by thermal changes may be 800–1000 ft., and that from rising strand line 700 or 800 ft. Thus, by a combination of both, about an additional 9000 ft. of sediments may be assigned to the basin, and the total thickness would be 21,000 ft.

Crustal collapse will fold these sediments into a great mountain system. To maintain the isostatic balance, the beds must be downfolded about six miles for every mile of upfolding. Tangential compressive stresses will be dominant, and the great amount of shortening which will occur in the geosynclinal prisms will give rise to intense crumpling, overturned folds, and thrust faults.

After the mountain system is moulded and crystal equilibrium established, the main movements will be vertical. Erosion of the high levels will disturb the isostatic balance and the adjustment will be upward. Final erosion of this mountain system will result in the isogeotherms being above normal, and on their return to normal the consequent shrinkage will give rise to a basin where once stood a mountain range. Thus, a mountain system which has been base-levelled can become the site of a new geosyncline merely as a result of temperature adjustments. A series of diagrams illustrates the course of events in the history of a mountain system.

The concluding section of this paper is devoted to the origin of a magma, and diagrams are also furnished showing the various stages in its formation. G. S. S.

1247.* Salem Oil-field Marion County, Illinois. H. H. Arnold, Jr. *Bull. Amer. Ass. Petrol. Geol.*, 1939, **23,** 1352–1373.—This paper deals particularly with the stratigraphy and structural geology of the Salem Oil-field.

A thick mantle of glacial material (40–60 ft.) covers the bed-rock of the area. From a drill-section—which is figured—the sequence of subsurface rocks shows approximately 1100 ft. of Pennsylvanian sediments represented by the McLeansboro, Carbondale, and Pottsville series. These consist of alternating shales and sandstones, together with thin limestones and coals.

The Pennsylvanian is followed by approximately 750 ft. of Mississippian sediments, of which about 680 ft. is represented by the Chester series, consisting of vari-coloured shales, sandstones, and crystalline limestones. The oldest rocks of the Mississippian reached by drilling were those of the Meramec group, the upper member of which is the St. Genevieve limestone.

Indications of early structural movements are evident in the Illinois basin, so much so that definite controlling influences were probably formed by these disturbances. Structural maps and cross sections of the Salem field are provided.

Production is obtained from the Mississippian, viz. the Chester series (Benoist Sand and Aux Vases Sand), and the Meramec (McClosky oolitic limestone). Oil has not been encountered in the Pennsylvanian rocks. The average daily production during June 1939 was 133,643 brl. and the total production for the entire pool (to July 1, 1939) was 20,080,000 brl.

Tables giving production data are included in the paper. G. S. S.

1248.* Jackson Eocene from Borings at Greenville, Mississippi. H. N. Fisk. *Bull. Amer. Ass. Petrol. Geol.*, 1939, **23**. 1393–1403.—The author describes a series of six middle Jackson Eocene cores from borings recently drilled in the alluvial-masked Mississippi River valley. The samples were taken at approximately 100-ft. depths along the water-front at Greenville. A cross-section is given showing thickness of Recent alluvium and position of samples in each boring.

From a study of the foraminifera found in the samples, it appears that the Jackson beds constitute the lower part of the *Textularia hockleyensis* zone and the upper part of the *Textularia dibollensis* zone. The junction of these zones has a westerly dip towards the Arkansas syncline, the axis of which is traceable to the south around the edge of the Monroe uplift into West Central Mississippi. G. S. S.

1249.* Illinois Geologic Trends Better Defined. W. V. Howard. *Oil Gas J.*, 26.10.39, **38** (24), 34.—Recent work has led to the general recognition of the importance of pre-Pennsylvanian structural movements. In addition to the Carlenville–Centralia and the parallel Ava–Dupo anticlinal trend, alignment along other north-west to south-east areas can be seen elsewhere in the basin. The subordinate north to south structural trend is becoming more apparent in the elongation and alignment of fields in this direction. The north-north-east to south-south-west trend is most evident in the south-eastern part of the basin in the Cione–Noble and Storms–Allendale trends.

The number of potential pay horizons has been increased, notably in the Chester series, where the Aux Vases, Renault, Tar Springs, Paint Creek, and Waltersburg sands have proved productive. The pre-Mississippian formations are disappointing, although the Devonian–Silurian (Niagaran) and Trenton have given provocative scattered production.

Details are given of wildcatting in the first nine months of 1939. Practically all the tests to pre-Mississippian horizons have been on the northern and western rims of the deep basin. Some of the new fields and the trends are briefly noted and a table gives the depths of the principal producing horizons. G. D. H.

1250.* Six Horizons Producing along Wabash Valley. K. A. Spitznagel and H. Moore. *Oil Gas J.*, 26.10.39, **38** (24), 54.—Nine oil-pools and one gas-well have been found in five months in the Wabash Valley. These run for 35 ml. and yield oil from the McClosky lime, Aux Vases sand, Cypress sand, Tar Springs sand, Waltersburg sand, and Biehl sand. The oil-fields of the Wabash Valley are only partly adjusted to structure. The Griffin trend is a series of highs aligned north-east to south-west. The producing horizons are lenticular. However, the McClosky lime production is entirely structural. The fields are yet too young for accurate estimates of recoveries, but the Cypress is put at 250 brl./acre-ft., Waltersburg 300 brl./acre-ft., McClosky 100–600 brl./acre-ft.

The Griffin pool yields oil from four horizons : the McClosky, 9 ft. thick ; the Cypress, 18 ft. thick ; the Waltersburg, 33 ft. thick ; and the Tar Springs, sand. 500 acres have been proved in the Waltersburg sand pool on Ribeyre Island. 45 wells have been completed in the Keensburg pool. Information is given about some of the smaller fields and a stratum contour map is included. G. D. H.

1251.* Reserves of Illinois Exceed 500,000,000 Barrels. H. F. Simons. *Oil Gas J.*, 26.10.39, **38** (24), 58.—Exploration during the past three years has revealed reserves of 495,132,000 brl. recoverable from nearly 56,000 acres, and during that period the production from the Illinois basin has been 70,237,173 brl. Most of the reserve lies in two major fields—Louden 182,000,000 brl., and Salem 200,000,000 brl. The former has produced 11,909,452 brl. and the latter 30,688,632 brl. of oil. Louden was discovered late in 1937, and Salem in midyear 1938. Both have over 1100 wells, and Louden is productive from the Cypress, Paint Creek, and Bethel sands, and Salem from the Benoist and Aux Vases sands and the McClosky lime.

The reserve estimate of 15,000,000 brl. for the Griffin area may have to be raised later, for all the pay horizons are discontinuous, rendering assessment dependent on detailed drilling. This area yields oil from the McClosky lime, the Cypress, Waltersburg, Tar Springs, and Biehl sands.

The Illinois basin is being depleted rather rapidly and it is unlikely that the present high discovery rate will be maintained. G. D. H.

1252.* Introduction to the Geology of North-west Peru and South-west Ecuador. A. Olsson. *Ann. off. Combust. liq.*, May 1939, **14**, 551–604.—The oil-fields of north-west Peru and western Ecuador are in the Tertiary zone along the Pacific coast. Outcropping beds range Upper Palæozoic to Recent. The oldest dated rocks are Pennsylvanian, and occur in the Amotape Range. These are overlain in places by Cretaceous limestones and shales, sandstones, and conglomerates of Aptian to Maestrichtian age. Representatives of the Trias, Jurassic, and Neocomian are known only in the south. Because of their economic importance, the Tertiaries have been studied in detail. Wide areas of Tertiary are covered by Quaternary tablazos. The stratigraphy is described in some detail. In the coastal region the Tertiary is 8000–10,000 m. thick, although there are wide variations in thickness. Lithologically the Tertiary consists of alternating shales, sandstones, and conglomerates, with only minor limestone beds.

The main structural features of north Peru are the Andean chain and Amotape Mountains, with the Pacific deep in the west. Between these are Tertiary deposits, folded and faulted. On the La Brea–Negritos anticline the main Peruvian oil-fields are developed.

Zorritos produced from the Miocene on a narrow block fault. At Cabo Blanco and Restin oil is obtained from the Eocene on a complexly faulted structure. The Lobitos field yields oil from a fold. The very extensive and important La Brea–Parinas field is on a large fractured dome, and derives oil from the Middle Eocene.

In the main, the geology of south-west Ecuador resembles that of Peru, but on the Santa Elena peninsula there are overturning, profound silicification and eruptive rocks. The Eocene rests unconformably on older disturbed beds. In the Oligocene are bituminous shales, but Miocene beds are unknown on the Santa Elena peninsula. There are many signs of oil, mainly in the zone of silicified and eruptive rocks. Eocene oil sands outcrop on the coast, and in this region is the main Eucadoran oil-field giving oil from the Eocene at depths of 200–800 m. G. D. H.

1253.* Oil-fields in Peru. J. E. Rassmuss. *Petrol. Z.*, 1939, **35** (32), 581–591.—A detailed geological and economical survey is given of the Peruvian oil-fields. The oil-field of northern Peru between Bayovor and Zorritos will continue to be of outstanding economic importance. A very rich field is situated in the Amazonas territory near Agua Caliente on the Rio Ucayali. In the neighbourhood of the oil-fields, this river has a width of 800 yd. and a depth of 8–20 ft., according to seasonal fluctuations. This field will, therefore, be of great importance in the future supply of Brazil, to which country the oil can be shipped in tankers. All other fields will never be of more than local value. E. W. S.

1254.* Geologic Structure and Oil Content of Syukeyevsko–Uleminski Rayon. A. M. Melnikov and V. F. Yakovlev. *Neft. Khoz.*, June 1939, **20** (6), 14–17.—The Syukeyevsko–Uleminski rayon is situated in the western area of the Tartar Republic on the right bank of the Volga river, 100 km. downstream of Kazan. The tectonic structure is discussed in detail, and conclusions are given from geophysical data available. Bitumen and sulphur are found on the earth surface at many places, the former being taken as an indication of oil-carrying strata at greater depths. The importance of thoroughly prospecting the rayon is stressed. L. R.

1255.* There is Oil in France. Anon. *Rev. Pétrolif.*, 28.7.39 (848), 986–987.—Bertrand has indicated the importance of the area along the northern margin of the Pyrenees in regard to oil prospects. Although about fifty wells were drilled in the Basses–Pyrenees and Landes, the absence of surface oil indications probably deterred searchers in Haute-Garonne. After various people had taken up concessions and renounced them, in 1938 the O.N.C.L. took up a concession of 49 sq. km. The first well found marls with thin sandstone bands intercalated down to 1430 m. Deeper there were Cenomanian limestones and conglomerates, which could form good reservoir rocks, under a thick, impermeable cover. At 1460 m. there was a gas flow, and a second one at 1520–1530 m., which gave 183,000 cu. metres/day. The presence of butane and higher homologues in the gas points to the possibility of its being associated with oil. G. D. H.

1256.* Important Discovery of Natural Gas in Haute-Garonne. Anon. *Rev. Pétrol.*, 21.7.39 (847), 955.—The Saint-Marcat anticline near Saint Gaudens (Haute-Garonne) was examined geophysically and by shallow borings. The first well was abandoned, and the second, possibly on the crest, had revealed a flow of gas which may well be associated with oil, at a depth of 1520–1530 m. An estimated production of 200,000 cu. metres/day was obtained.

A well is to be drilled to test the flank at Pic Saint-Loup, near Montpellier.

Gas has also been found at Lussat, Pont-du-Chateau, Chamailliere, Espirat, Macholles, Puy de la Poix, Puy Crouelle, etc. The best-known occurrence was at Vaux, where a well in 1920 gave gas under a pressure of 15 kilos/cm.². G. D. H.

1257.* Deposit of Bentonite in Mendoza. Decree. Anon. *Bol. Inform. Petroleras*, June 1939, XVI (178), 36.—Decree of 6th May, 1939, granting a concession to the Y.P.F. to work a bentonite deposit in Potrerillos (Lujan). H. I. L.

1258.* The Celluloses in Algæ. G. Viel. *Ann. Off. Combust. liq.*, May 1939, **14**, 531–550.—The findings of the previous investigations of the constituents of algæ are briefly summarized. In the new work cellulose from *Fucus vesiculosus*, *F. serratus*, *Laminaria saccharina*, and *L. cloustoni* was examined. The method of preparation, the properties, and the proportions of the cellulose in these algæ are described. These celluloses are cellular, and in some cases fibrous. On hydrolysis they gave quantities of glucose of the order which should be given by celluloses.

These celluloses were heated to 1000° C. in 100° C. stages, 15 min. being allowed at each stage and 1 hr. at the final temperature. The volumes of gas evolved per gram have been plotted for the temperature stages, and the residual coke and heavy products were weighed. The curves obtained resemble those for vegetable celluloses. There is a maximum in the range 200–400° C., and a less prominent maximum at 700° C. Carbon dioxide and carbon monoxide were evolved most abundantly at 300–400° C., whilst methane appeared mainly at 500° C., except in the case of *Fucus serratus*, which has a maximum at 600° C. Hydrogen was evolved most strongly at 700° C.

G. D. H.

Geophysics.

1259.* Hydrological Exploration in Octyabrneft Fields. V. P. Yakovlev. *Neft. Khoz.*, March 1939, **20** (3), 25–30.—A report is given on work carried out to confirm the assertion that micro-oscillations of fluid levels in abandoned and temporarily shut-down wells are of exceptional importance, providing much information on capacity, etc., of the strata under exploitation which is otherwise impossible to obtain.

By the method indicated, numerous data have been obtained on the reservoir under examination which enable all necessary forecasts to be made on its future behaviour under all possible conditions, beginning with changes in hydraulic pressure owing to additional drilling of any number of wells, to the height of microstatic and dynamic levels in the oil wells of the reservoir under any rate of withdrawal and for any number of wells given.

From another, considerably larger, reservoir much less data were obtained.

The difference between the present method and those indicated by Hurst and Muskat is that the latter base their conclusions on information, obtained by other means, on the geological formation of the Woodbine layer, to determine its hydrodynamic properties, whilst in the method described, from the hydrodynamic manifestations of the sub-surface system information is obtained not only regarding its capacity, resistance, and other hydrodynamic properties, but also regarding the geological structure of the reservoir. L. R.

1260.* Electrical Measurements in Bore-holes. C. E. Ripamonte. *Bol. Inform. Petroleras*, June 1939, XVI (178), 56–59.—Examination of the nature of the substrata surrounding the bore-hole is done electrically; the measurements that are most commonly made are those showing the specific resistance and those indicating the permeability of the soil at the measuring point. Various bridge circuits are described for use with direct current, and reference is also made to others for use with alternating current. H. I. L.

Drilling.

1261.* Unitized Portable Skid for Boiler-Feed Pumps. Anon. *Oil Wkly.*, 5.6.39., **93** (13), 50.—A description is given of a simplified skid for boiler-feed pumps which requires only a few minutes to break the joint and winch the whole unit on a truck for transportation to another location. This type of assembly is rapidly replacing the older units at all boiler batteries in Santa Fe Springs, Monte Bello, and in other fields where there are company-owned drilling tools. A. H. N.

1262.* Throw Away a Few Feet of Rope and Save Several Hundred. Anon. *Oil Wkly.*, 26.6.39, **94** (3), 30.—A discussion on safely handling rope to yield maximum working life and efficiency. A. H. N.

1263.* Number of 10,000-ft. Tests Increasing Rapidly. W. L. Baker. *Oil Wkly*, 31.7.39, **94** (8), 59.—An interesting compilation of record-breaking depth tests is presented in this paper, which is one of a series of articles dealing with deep drilling published in this issue of the *Oil Weekly.* The paper is followed immediately by a second on the same lines. A. H. N.

1264.* Improved Practices Permit High-Speed Deep Drilling. B. Mills. *Oil Wkly*, 31.7.39, **94** (8) 66.—This is the third paper in the series of articles dealing with deep drilling published in this issue of the *Oil Wkly.* The record for deep drilling has been nearly doubled in the last 12 years. In 1927 the deepest penetration was 8046 ft.; to-day it is 15,004 ft. The ability to reach such depths is indicative of the vast improvements in technique and equipment.

The speed of drilling is particularly noteworthy, the speeds of to-day being about three times those of but a short time ago. Higher rotating speeds, and increase in weight on bit, the use of long and heavy collars to control the weight on the bit, accurate and constant mud control and controlled vertical drilling, and the adaptation of high steam pressures are all contributory factors which are analysed in detail by the author. Typical practices in various fields are given. A. H. N.

1265.* New Cementing Problems Created by Deep Drilling being Overcome Rapidly. B. Mills. *Oil Wkly*, 31.7.39, **94** (8), 110.—The conclusion of this detailed discussion on new cementing problems encountered in deep wells may be summarized as follows:—

(1) Regardless of the safety factor provided by equipment and materials used on the job, every possible hazard that can be anticipated should be eliminated with as little delay as possible.

(2) The well bore should be conditioned properly to receive casing and provide space for satisfactory volume of cement back of the casing.

(3) The rotary mud should receive constant attention during the entire period of casing and cementation. The viscosity should be maintained as low as is consistent with the necessary density.

(4) The cement used should remain pumpable well beyond the time allotted for pumping it in place. Anything may happen to upset the time-estimate.

(5) Cement and equipment should be selected on the basis of quality and adaptability to conditions.

(6) From 50 to 100% more cement should be used than the amount shown by calculation. A. H. N.

1266.* Deeper Drilling Requires Closer Mud Control. H. H. Farnham. *Oil Wkly*, 31.7.39, **94** (8), 126.—Inherent with drilling to greater depths, two factors are encountered which make it vitally essential that closer attention be given to the control of the properties of mud than is necessary in shallower wells :

(1) High temperatures are encountered, which may be between 200° F. and 240° F.
(2) The higher hydrostatic head of the mud means loss of mud at a fast rate when low-pressure, high-permeability sands are encountered.

These two factors are discussed, together with high-pressure filtration test apparatus, and typical examples of mud conditions in various fields in U.S.A. utilizing deep drilling equipment and methods are cited. Details are given of weights, viscosities, filtrates, cake thicknesses, and salt contents of the typical muds, together with methods of attaining these results. A. H. N.

1267.* Closer Determination of Pressures Would Help in Deep Operations. Anon. *Oil Wkly*, 31.7.39, **94** (8), 78.—Pressures maintain a fairly true rate of increase down to about 7500 ft., but below that level variations become more frequent and pronounced, thereby accentuating the hazard of blowouts on deep drilling. The paper deals with normal high pressures and freakish conditions encountered in deep drilling.

A. H. N.

1268.* Relation of Preserved Wood to Deep Marine Operations. R. S. Manley. *Oil Wkly*, 14.8.39, **94** (10), 22.—The treatment of wood intended for derrick foundations in marine oil-well drilling is discussed. In locations off the coast in salt water the usual American practice is to have the timbers impregnated with at least 20 lb. of creosote per cubic foot if they are to be immersed in the water, and with 12 lb. per cubic foot if not actually submerged. For fresh-water locations in marshes or lakes, where the water becomes only slightly brackish part of the time, 16 lb. per cubic foot of wood for submerged timbers and 12 lb. if not submerged are considered to furnish ample protection. A. H. N.

1269.* Cement for Oil Wells. W. W. Robinson. *Oil Wkly*, 21.8.39, **94** (11), 20.—Paper presented before American Petroleum Institute.—The paper presents a compilation of test methods being used to evaluate cement for use in oil wells. These special procedures, which have been developed in the industry because standard test methods are not applicable, are given in detail, with purpose, procedure, interpretation, and practical application of test data. Results of tests covering several cements used in oil wells are shown. There is a definite need for standardization of methods for testing cements used in oil wells, and it is proposed that an American Petroleum Institute code be formed. In this, the first part of the paper, sampling, determination of standard slurry fluidity, and thickening-time tests are discussed. A. H. N.

1270.* Cement for Oil Wells. W. W. Robinson. *Oil Wkly*, 28.8.39, **94** (12), 18.—This is the second part of the paper presented by Mr. Robinson before the A.P.I. (*cf.* Abstract **1269**). The author details procedures for consistometry, tensile strength, Foam tests, and physical properties testing of cements for use in oil wells. A. H. N.

1271.* Results of Australian Drilling Discouraging. Anon. *Oil Wkly*, 21.8.39, **94** (11), 48.—Although the Federal and State governments of Australia are prone to do what they can to encourage prospecting for oil in that commonwealth, results at the end of 1938 were not very encouraging. Details of 38 wells are given in tabular form.

A. H. N.

1272.* Enormous Assembly used on Gulf Coast Rig. N. Williams. *Oil Gas J.*, 22.6.39, **38** (6), 54.—Interest is shown in the production potentialities of the old Jefferson Island salt dome due to a deep test being drilled on the extreme north flank. For this test one of the largest, most modern, and most complete assemblies of drilling equipment on the Gulf coast is being used. The rig embodies a 10-in. draw-works—the largest and newest size made. It has a 20½-in. high-speed oil-bath rotary driven by a separate 9 by 8 in. horizontal steam engine; two 20-in. mud pumps; an 18-in. mud-mixing pump with a desander and a degasser; a 990,000-lb. load, 136-ft. steel derrick with a 36-ft. base; a 700-000-lb. six-sheave travelling block, and a seven-sheave crown; four 130-h.p., 350-lb. working-pressure superheated boilers; a boiler feed-water installation, and two generators to supply lighting and power requirements.

A feature is the horizontal steam-rotary-drive engine mounted below the derrick floor to provide greater working space and room on the floor. The engine is hooked up not only to the rotary immediately above, but also to the coring reel and to the main draw-works, which can be run by the rotary engine in an emergency.

For the drilling hook-ups, provided to meet any emergency, two complete blow-out-preventer assemblies have been provided, mounted one above the other, with a 13⅜-in. master gate-valve below each.

Other details are given about the mud system and the drilling programme.

A. H. N.

1273.* Survey of Drilling Equipment Reveals Economic and Operating Data. H. F. Simons. *Oil Gas J.*, 29.6.39, **38** (7), 34.—The data, obtained by means of a survey, based on thousands of questionnaires sent out to drilling contractors and drilling departments of oil companies all over the U.S.A., are summarized. Diagrams are given to illustrate the statistics. The paper deals mostly with the economics of drilling, but an interesting item is that the total number of rotary rigs was 47·2%, whilst cable-tool rigs accounted for 52·8% of the total number of rigs employed. Although no figures are available, it is almost certain that the greater share of footage is drilled by the rotary. More than half the rotary rigs use steam for the prime mover.

A. H. N.

1274.* Positive Vertical Drilling Cuts Costs and Saves Time. Anon. *Oil Gas J.*, 29.6.39, **38** (7), 40.—Positive vertical drilling is generally being accepted as beneficial, instead of being an addition to the cost of drilling. The factors to be studied are the conditions in the hole, the re-design of the drill stem, accurate determination of the deviation of the hole, and the selection of proper rotary speeds and weights on the bit. When rotary drilling in the Seminole field of Oklohoma yielded erratic results in the geological departments due to deviation of hole, acid-bottle tests were introduced, and it was shown that wells deviated by as much as 27°. For an average deviation of 5° the loss of footage on the 4400-ft. hole would be 16·72 ft.—enough to upset any geological map drawn on the information.

The costs chargeable to crooked holes come under two classes: direct and indirect. The items under each class are many and varied. Sometimes it is almost impossible to estimate the indirect costs due to crooked holes, such as increase in wear on drill pipe and breakoffs which have been accelerated by continuous bending and flexing, and increased difficulties in fishing jobs. Again, the bits are designed to yield maximum efficiency in a vertical hole, and hence will not be efficient in crooked holes. But no mathematical formula can give the loss in efficiency, as the factors involved are many and complex.

It is now accepted that a vertical hole is drilled as quickly and often quicker than a crooked hole, provided the proper equipment and technique are employed.

Two main factors affect the problem: geological conditions, and mechanical design and operation of the equipment. The former is unalterable, but, where adverse, can be circumvented by altering the second factor.

When the bit moves from a soft to a hard formation in an inclined structure it tends to move down dip, and vice versa. Rapid rotation and the application of as little weight as possible on the bit keep the hole vertical, but the time taken is prohibitive.

It is shown that so long as the drilling pipe is stable—*i.e.*, a vertical line passing through its centre of gravity falls within the base of the bit—the hole will be vertical. The C.G. of the pipe rises with increased weight on the bit. By increasing the weight of the drill collar, the centre of gravity is kept low and the ideal is approached. Some operators use 400-ft. drill collars. Addition of reamers or guides to centralize the collar in the hole confers further advantages.

Deviation tests should be taken continuously. Acid-bottle and ink-siphon inclinometers, together with chart-recording instruments of the sharp-pointed plumb bob and photographic types, are briefly described.

The readings should be taken often, and in a strange locality at every 100 ft. As long as the elements producing deviation are not severe, straightening is easily accomplished by removing weight from the bit and increasing the drilling speed of the table. Beyond a "critical angle" straightening becomes much more difficult. Plugging back may be resorted to, a whipstock may be run, or the bottom may be enlarged by a wall scraper to allow the collar to assume a vertical position. A. H. N.

1275.* Low Cost of Drilling for Michigan Operators. H. F. Simons. *Oil Gas J.*, 6.7.39, **38** (8), 12.—This is the second of two articles that discuss the present petroleum situation in Michigan. The geology and prospects of the fields are discussed in the first part of the paper.

Most of the drilling is done with cable tools, either standard rigs or spudding machines being used. There are only a few rotaries at work in the area, and these are used mostly for the drilling of drift and the Pennsylvanian formations. Using a rotary rig to drill the upper hole saves two strings of pipe in the fields where the Michigan stray is productive, as this formation must be sealed off. In the shallow fields three strings of pipe are run, 10 in. being driven through the drift and $8\frac{1}{4}$ in. used to shut off the cold-water formation. Either $5\frac{3}{16}$ or $6\frac{5}{8}$ is used for an oil string in most Michigan fields.

A producing well can be drilled to 1550 ft., cased, treated, and equipped with pumps, rods, and tubing for $7500 ; lift costs average 15 cents/brl. for the average recovery of 27,000 brl. for a 10-acre spaced well. A. H. N.

1276.* Utilize New Type of Mud Circulating System. N. Williams. *Oil Gas J.*, 13.7.39, **38** (9), 42.—A new mud system for drilling deep, high-pressure tests on the Gulf Coast gives (1) increased efficiency in mud-pump operation, (2) greater flexibility in conditioning and control of drilling fluid, with maximum protection against contamination, and (3) substantial savings in expenditure for mud and weighting materials. The new mud system embodies gravity flow of the drilling fluid to pump suction. The derrick floor has to be elevated to accommodate the greater height needed for the mud return line from the well to the ditch, with the advantage of increased clearance and working space under the floor, facilitating installation of the large drilling and blow-out preventer assemblies being used in the controlling of deep, high-pressure wells. A. H. N.

1277.* Drilling-Time Data in Rotary Practice. T. C. Hiestand. *Oil Gas J.*, 13.7.39, **38** (9), 55.—Drilling records have been used for some time, but the method has not been fully utilized until recently. Drilling-time data assist (1) in fuller recovery of cores, (2) in avoidance of undue loss of time in running on dull bits, and (3) permit the correction for sample lag, so that the log of the well checks with electrical and geothermal charts. The controlled drilling possible with modern rotary rigs, together with the use of the geolograph, making drilling-time records dependable for interpreting accurate depths of porous formations on the rig floor. This in turn allows the geologists, on drilling wells, to recommend appropriate testing of all zones where oil and gas occur. Six typical well cases are discussed. A. H. N.

1278.* Drilling-Time Data in Rotary Practice. P. B. Nicholls. *Oil Gas J.*, 20.7.39, **38** (10), 40.—Manual recording of drilling time has been used quite successfully, but the human element enters largely into the accuracy of the data. The information has been restricted chiefly to the depths where production is expected, and has not been kept for the majority of rotary operations. The paper describes the geolograph, an instrument which automatically records the rate of penetration, foot by foot, together with such related information as time out for repairs, round trips, etc. A diagram is given of the hook-up, and illustrates the manner in which the motion of the tools is recorded. Typical drilling changes in rates of penetration, together with information as to when the tools are on bottom, when drill-pipe connections are made, samples circulated, etc., are given in a typical chart. A. H. N.

1279.* Drilling Practice Committee. Anon. *Oil Gas J.*, 20.7.39, **38** (10), 50.—A report of the Drilling Practice Committee of the eastern division of the American Petroleum Institute is given. This report deals primarily with the costs and improvements in rotary and cable-tool drilling in the Illinois and West Virginian areas.
A. H. N.

1280.* Complete 12,000-Foot Test in Louisiana Under Contract. N. Williams. *Oil Gas J.*, 3.8.39, **38** (12), 50.—The recent drilling on the Louisiana Gulf Coast of two wells under contract to 12,000 ft. reflects the greater confidence and certainty with which deeper exploration and development are being undertaken. Numerous tests have been drilled to that depth and to much lower levels, but these are the first instances in which a drilling contractor has assumed the risk attendant upon drilling to and completing a well at such a depth. The first well to be completed was the Gulf Refining Company's No. 1 Grandison in La Fourche Parish, coastal Louisiana. A description is given of the equipment used and operations in connection with the drilling of this test. A. H. N.

1281.* Skidding Derricks in the Permian Basin. D. H. Stormont. *Oil Gas J.*, 10.8.39, **38** (14), 44.—Where the topography is favourable and the distance is short, the moving of derricks to new sites is done more economically by skidding than by dismantling and re-erecting. Factors to be taken into account when derrick skidding is under consideration are discussed and current practices employed in skidding operations in the Permian basin of West Texas and New Mexico are described. A. H. N.

1282.* Directionally Drilled Wells Drain Oil from under Lake Centralia. H. F. Simons. *Oil Gas J.*, 31.8.39, **38** (16), 34.—By using modern methods of directional drilling, the formations under this Illinois lake are being effectively drained. A complicating factor met by the operators was the shallow depths of the wells, and the consequent high angles of deflection. It was feared that deflections of approximately 400 ft. might be not only economically prohibitive, but physically impossible to drill. For this reason it was decided to drill one of the longest deflections on the original attempt as a test job for the method.

The details and results of the drilling are given together with charts showing a horizontal and a vertical plan of the completed well. A. H. N.

1283.* Steel-Fabricated, Arc-Welded Field Slush Pump. W. C. Russell and L. W. Stahl. *Oil Gas J.*, 31.8.39, **38** (16), 40.—Deviating from the usual practices, the Emsco Derrick & Equipment Co. have developed a slush pump made up entirely of steel structural shapes and plates, steel forgings and castings welded together at the juncture of all parts. An exhaustive description, accompanied by many illustrations, is given of the many parts composing this mechanism and of the method of fabrication.
A. H. N.

1284.* The First Horizontal Oil Well. L. Ranney. *Petrol. Eng.*, June 1939, **10** (9), 25.—A detailed account is given of the experiment conducted in horizontal drilling in a shallow oil sand. It is a description of the first horizontal oil well. A report is also given of a radial pattern of wells drilled horizontally from a central chamber in a property developed by the Ranney method. The manner and effects of shots are detailed and illustrated by graphs. A. H. N.

1285.* Californian Deep Drilling. W. A. Sawdon. *Petrol. Engr.*, July 1939, **10** (11), 27.—Greater advance has probably been made in the technique of high-speed rotation in drilling deep wells in the San Joaquin Valley, California, than in any other area in the world. Rotating speeds of 400 r.p.m. and greater have become more or less common in the Rio Bravo field, and a rotation speed of 600 r.p.m. has been employed in one operation in nearby territory. This speed was used mainly in testing, and only a small amount of hole was actually made at 600 r.p.m. A. H. N.

1286.* Heavy-Duty Drilling Equipment Required in Magnolia Field, Arkansas. H. L. Flood. *Petrol. Engr.*, Aug. 1939, **10** (12), 23.—Orderly development has resulted in 45 producers on 40-acre spacing. The author gives the early history of the field; details and justifications of the heavy equipment used; casing programmes and drilling speeds. The average depth of hole made per day is 190 ft. A. H. N.

1287.* Mud Analysis Used to Log Wells While Drilling. W. A. Sawdon. *Petrol. Engr*, Aug. 1939, **10** (12), 84.—Test equipment mounted compactly in a trailer is used to detect oil, gas, or salt water in formation being drilled. It also provides warning of wash-outs in advance of pipe failure. A. H. N.

1288.* Large Drilling Equipment Tested on Two Wells in California. W. A. Sawdon. *Petrol. Engr*, Aug. 1939, **10** (12), 95.—This long paper deals with the results of testing a heavy set of drilling equipment, which was designed for drilling 18,000–20,000 ft. depths, under rigid control to depths of 11,500 ft. in each of two holes. Full details are given. A. H. N.

1289. Merits of Zinc-Coating Threads of Drilling Pipe Joints. S. G. Znaichenko. *Azerb. neft. Khoz.*, December 1938, **18** (12), 29–30.—With increasing drilling depths, and consequently increased stresses, there is a rising tendency for the threads of drilling pipe joints to seize, resulting in reduced life of drill pipes. The effect of zinc-coating pipe threads to eliminate seizure has been examined.

It has been found that zinc-coating of drill-pipe joints remains effective even after prolonged service, whereas the graphite lubricant used heretofore always tended to be displaced or to harden and, consequently, could not efficiently prevent seizure or corrosion. In view of the low cost and ease of application, zinc-coating of the threaded pipe ends and pipe joints is strongly recommended. L. R.

1290. Cement with Hydraulic Admixtures for Oil-Well Cementing. N. A. Lutsenko. *Azerb. neft. Khoz.*, December 1938, **18** (12), 25–29.—Tests have been carried out with cement slurries containing varying proportions of "acid-hydraulic" admixtures (consisting mainly of silica and alumina).

The work has been divided into three parts: (1) examination of cement properties at normal and increased temperature, (2) effect of agitation on cement properties at normal temperature, and (3) determination of the temperature which develops during hardening.

It has been found that, in principle, Portland cement with acid-hydraulic admixtures may be used for cementing operations, giving great stability and resistance of the cement ring against the action of chemically active waters. To cement from the Kaspi factory 10% pumice stone may be added when used for shallow wells, for deep wells 20%. To cement of the Baku factory for shallow wells 15% pumice stone may be admixed, for deep wells 20% or more.

All types of cement examined, starting from the first hour of agitation of the slurry, decrease in physical strength with increasing intervals during which the slurry is in the dynamic state. The temperature developed during setting and hardening of cement containing pumice stone is sufficient for measuring electrothermically the height of cement reached in the space behind the casing. L. R.

1291. Prevention of Accidents in Drilling. V. Mussini. *Bol. Inform. Petroleras*, June 1939, XVI (178), 84–88.—Detailed description, with illustrations, of the points requiring attention in order to prevent accidents when carrying out work on the towers and drilling operations generally. H. I. L.

1292.* Transport of Fully Equipped Drilling Towers at Comodoro Rivadavia. H. Giraldez. *Bol. Inform. Petroleras*, June 1939, XVI (178), 45–52.—Illustrated description of movement of fully equipped drilling tower to a new emplacement. The tower was left in its normal vertical position and mounted on a skid of special construction. The relatively flat terrain made this possible. H. I. L.

1293. Transport of Heavy Weights at Comodoro Rivadavia. H. Giraldez. *Bol. Inform. Petroleras*, June 1939, XVI (178), 53–55.—Article illustrating the heavy weights that have to be transported whenever plant has to be set up at a fresh borehole in the field.
H. I. L.

1294. Drilling Patents. M. Hokanson. U.S.P. 2,156,287, 2.5.39. Appl. 21.11.36. Drill bit with substantially circular cutting edges.

R. Cross and M. F. Cross. U.S.P. 2,156,333, 2.5.39. Appl. 6.3.37. A method for cleaning oil-well drilling fluids in which screens and a centrifugal pump are employed to eliminate all the cuttings and solid particles before recycling the mud.

R. L. Fluellen. U.S.P. 2,156,384, 2.5.39. Appl. 28.1.37. A slip for holding pipes.

J. A. Zublin. U.S.P. 2,156,444, 2.5.39. Appl. 29.8.38. Rotary reamer.

C. P. Walker. U.S.P. 2,156,519, 2.5.39. Appl. 7.9.37. Means for measuring the location of obstructions in wells by creating a pressure wave and recording the echoes.

A. C. Catland. U.S.P. 2,156,676, 2.5.39. Appl. 5.11.37. A rotary bit with cutters mounted on ball bearings.

R. E. Fulkerson. U.S.P. 2,156,939, 2.5.39. Appl. 29.3.37. A well packer.

M. Miller and L. N. Davis. U.S.P. 2,157,493, 9.5.39. Appl. 29.4.38. Well scraper.

R. R. Bloss. U.S.P. 2,157,728, 9.5.39. Appl. 22.11.37. Speed-up and speed-down for rotaries.

H. W. Buschman. U.S.P. 2,157,951, 9.5.39. Appl. 24.9.38. Well drilling cable control.

H. E. Grau and R. K. Hertel. U.S.P. 2,158,232, 16.5.39. Appl. 6.12.37. Drilling hook with ball bearing and spring elements.

J. J. McCann. U.S.P. 2,158,243, 16.5.39. Appl. 3.3.38. Casing cutter of the knife cutter type.

F. Kinzbach. U.S.P. 2,158,329, 16.5.39. Appl. 6.8.37. Whip stock for deflecting milling tools.

J. G. Dyer. U.S.P. 2,158,356, 16.5.39. Appl. 16.9.35. A drill pipe coupling means.

S. W. Long. U.S.P. 2,158,372 and 2,158,373, 16.5.39. Appl. 3.9.36. Spring hook.

C. H. Collett and A. R. Thompson. U.S.P. 2,158,406, 16.5.39. Appl. 17.5.37. Liner setter.

J. T. Ashcroft. U.S.P. 2,158,814, 16.5.39. Appl. 24.1.38. A fishing tool for drill stems.
A. H. N.

Production.

1295.* Application of Vacuum to Pumping Wells. W. A. Sawdon. *Petrol. Engr*, June 1939, **10** (9), 31.—One benefit resulting from the use of high vacuum in pumping wells in several fields of California has been an increase in the gasoline content of the gas produced. Some operators believe vacuum operation results in increased oil production also. Others, whose records show no increase of production, realize the necessity of protecting their production by applying a vacuum as high as that used on adjacent wells.

The extremely close spacing of wells in the area has a definite influence on the amount of vacuum carried. The need to apply equal vacuum to neighbouring wells to effect a so-called " protection " was demonstrated when one well was put under atmospheric pressure to allow a clear-out. No fluid was found at the bottom of the well.

The functioning of vacuum pumps is given. A. H. N.

1296.* Production Methods in the Rodessa Field (Part 2 concluded). T. H. Gibbs. *Petrol. Engr*, June, 1939, **10** (9) 35.—It is estimated that 30% of the wells in the field produce by artificial lift. Of this number 85% produce by gas-lift and 15% by pumping. The latest type of gas-lift to receive wide acceptance in the field is the one known as the surface-controlled wire-line-operated type, which is described in detail in the paper. It is an intermitted gas-lift system where the operating valve is worked from the surface at regular intervals by means of a gas-turbine-driven wire-line hoist.

Pumping units and rigs are described in brief, and they present no unusual features. Sucker-rod troubles have not been excessive, and in the choice of rods the properties sought are great shock and fatigue resistance, ductility, toughness, and resistance to corrosion.

While hydraulic pumping is still in its experimental stage in this field, due to excessive corrosiveness of the water, two installations have already proved successful. A description of a hydraulic unit is given. It consists, in brief, of a pump-and-engine unit that is lowered in the well and a hydraulic power unit that is run from the surface. Clean oil pumped to the hydraulic engine actuates the pump.

Emulsion treatment is an easy task in the Rodessa field. The most popular methods are settling-tanks and heat, settling-tanks and chemical without heat, and chemical treatment without the use of heat or settling-tank.

Many attempts have been made to exclude bottom water or edge water, without appreciable success. Squeeze jobs have been satisfactory only in few wells—laboratory tests have shown that changes in volume as great as 30% can result from squeezing water from the cement into the low-permeability limestone formation, and thus misleading the operators as to the success of the operation.

Acidizing has been of great value to the field. It is used also in workover and clean-out operations. A departure from the customary two-pump oil-flush acidizing procedure is the use of gas to force the acid into the formation, thus preventing killing the well.

The paper also discusses plug-back operations, gun perforating, salt-water disposal, and the use of vacuum traps or blow-cases to lower separator pressure, with consequent increase in gasloine recovery. A. H. N.

1297.* Economic Study of Electric Power in Oil-Well and Pipe-Line Pumping Service. W. H. Stueve. *Petrol. Engr*, June 1939, **10** (9), 89.—Past performance records of oil-producing and pipe-line companies proved valuable in determining the merit of generating electric-power by the consumer, or of purchasing it for operations in oil pumping. In the choice of the electric-power source for oil-well pumping several factors must be considered. Some of the more important variables may be enumerated as follows :—

(*a*) Size and characteristics of the contemplated load.
(*b*) Nature and amount of fuel available and its cost or value.
(*c*) Possibility of water encroachment in the producing horizon.
(*d*) Probable decline of production in the specific oil-field.

These considerations are made in the paper, and a detailed study is also given of actual cases to illustrate the various points. A. H. N.

1298.* Electric Power Plays Important Rôle in Water Flooding of Nowata Field. J. D. Robertson. *Petrol. Engr*, June 1939, **10** (9), 109.—Of the 5200 acres under water-flood in Nowata field, Oklahoma, 3500 acres are being produced with the aid of electric power. The many problems arising in the various stages of water-flooding

operations are discussed. Tests are detailed, and the results are given in tabular and graphical forms. These should be helpful to operators planning a new project.
A. H. N.

1299.* Purchased Electric Power Section Symposium. J. E. Elliott and others. *Petrol. Engr*, June 1939, **10** (9), 131–140.—Three papers in this section deal with the application of electric power in oil-fields. The first, by J. E. Elliott, deals with East Texas and Rodessa Fields, and shows that proration of production has prompted combination billing for scattered meters and that demand rate permits low unit cost on high load factor. The second, by K. C. Johnson, deals with operations in Arkansas, where a total of 10,000 h.p. is distributed in oil-well and pipe-line pumping and refinery operations. The third, by N. W. Beaudreau, deals with South-west Texas oil-well pumping utilizing electric power which proves its worth in pumping wells situated in remote territory, where minimum attention permits pumpers to cover wide areas.
A. H. N.

1300.* Fundamental Considerations of Recycling in Condensate Production. E. Kaye. *Petrol. Engr*, Aug. 1939, **10** (12), 29.—In 1938, out of 111 new pools discovered in Texas, 27 were of the gas-distillate type. Careful consideration of all factors involved in recycling in such pools should safeguard against uneconomical installations. Such considerations are made in the paper dealing with the objectives and requisites of recyclings and with various recovery processes. The paper ends with a discussion of future developments.
A. H. N.

1301.* Hot-Water Heater for Preventing Freeze-Offs of Large-Volume Gas Wells. R. W. Parker. *Petrol. Engr*, Aug. 1939, **10** (12), 109–110.—A plan of the layout, and description of an installation in Cement Field, Oklahoma, is given. It is claimed that fire hazard that accompanies open-flame burning is thereby eliminated.
A. H. N.

1302.* Effect of Design and Direction of Rotation on Peak Torque of Pumping Unit. K. N. Mills and E. A. Olsovsky. *Petrol. Engr*, Aug. 1939, **10** (12), 113.—Torque capacity of a specific unit will vary with the direction of rotation, therefore both values should be known. This paper, which deals with torque and direction of rotation, is amply illustrated with figures, graphs, and tables to indicate how the geometry of a pumping unit, together with its direction of rotation, control the magnitude of the peak torque imposed on the reducer. It is argued that each unit should carry a torque factor on its name-plate, which would enable the user to determine the true torque capacity of the unit assembly when the crank rotates in either direction. A. H. N.

1303.* West Montebello Oil-field. H. M. Preston and V. L. King. *Petrol. World* (L.A.), Sept. 1939, **36** (9), 19–25.—The authors give the location and history, the operating conditions, and completion practice of the West Montebello field. A map and a section of the field illustrate the stratography and the five oil zones which form the productive regions up to date. The productivity of the five zones increases downward, the lowest being the most extensive. The present daily potential of the field is 40,000 brl.
A. H. N.

1304.* New Reserves in San Joaquin Valley. Anon. *Petrol. World* (L.A.), Sept. 1939, **36** (9), 28.—Gulf Oil Company's discovery of a second oil-field and new extensions to the existing fields will provide California with large additional supplies of crude oil for the future. The paper is a report on the new reserves at various localities in the new fields.
A. H. N.

1305.* Acid-soluble Liners for Oil-wells. Anon. *Petrol. World*, Sept. 1939, **36** (9), 68.—A new magnesium alloy pipe is being used as a removable liner by setting it as a section of casing and then removing it with the aid of inhibited hydrochloric acid. A wide variety of uses is described.
A. H. N.

1306.* Operating Practices Reduce Smackover Lift Costs. B. Mills. *Oil Wkly*, 12.6.39, **94** (1), 13.—Four types of wells exist in the Smackover field. The first pumps 50 brl. of oil and from a trace to a few barrels of water. The second pumps 25 brl. of oil and 100 brl. of water. The third pumps 25 brl. of oil and 500 brl. of water. The fourth pumps 10 brl. of oil and 1000 brl. of water. The average production of the field is 17,000 brl. of oil and 500,000 brl. of water daily. The present hard fight which faces the producers is directly due to poor drilling and production practices of 10 and 15 years ago. Hundreds of wells were drilled in a haphazard manner, blow-outs were numerous, over-production of large wells was rampant, and poor logs were in most cases worse than none.

The present oil reserve at Smackover is estimated at 60,000,000 brl., and attempts are being made to reduce lifting costs, the improvement being effected in equipment and methods where the greatest troubles arise rather than in a radical departure from former operating practices.

A travelling valve with a combination of cups and rings has been used to advantage in several wells. The composition rings are placed on the bottom of the valve and the cups on top. The rings are pre-swelled in oil, on the surface.

Troubles from sucker-rod failures above the 1000-ft. level have been traced to overloading, and the present practice is to use about 1000 ft. of $\frac{7}{8}$-in. sucker rods on top and from 1300 to 1600 ft. of $\frac{3}{4}$-in. rods on the bottom. The trouble is solved by following this practice and keeping a continuous check on handling the tubings properly and seeing that the threads are free from pits and imperfections.

" Pounding " in wells on the downstroke of the pump has been virtually eliminated by the installation of relief valves, which allow air into the lead lines on each stroke of the pump.

Constant studies of loads, strokes, vacuum requirements, submergence, reconditioning of wells, and equipment selection is helping in a hard fight to reduce costs. Corrosion trouble with sucker rods is severe, but it has been found that such troubles are particularly great with overloaded rods. Minor nicks and imperfections in rods which are working under conditions conducive to fatigue have often caused failures. Substitution of proper size for the rods and constant examination have eliminated the greater part of the troubles.

Alloy rods give better service.

Loose sand troubles have been reduced considerably by a study of fluid conditions, regulation of stroke, and the use of proper pumps. A. H. N.

1307.* Exception to Rule 37 (Relating to Oil-well Spacing). I. I. Gardescu. *Oil Wkly*, 12.6.39, **94** (1), 32.—The paper deals specifically with conditions in the East Texas fields, but is of general interest, in that a discussion is given of the merits and demerits of close spacing. It is shown that too close spacing is inefficient due to :

(1) too rapid lowering of reservoir pressure ;

(2) the claim of close-spacing advocates that energy is wasted in friction in long journeys of oil is not valid, as the rates of flow are extremely small beyond the immediate vicinity of the well ;

(3) water by-pass is more severe in close spacing, as there is a smaller pressure differential in the reservoir, since the average pressure difference between reservoir and producing well is distributed over larger areas, in the case of wide spacing within economic limits ;

(4) the danger of water contamination from upper sands is greater the greater the density of wells per unit area.

These remarks apply particularly to fields—such as those of East Texas—where evidence is forthcoming against the existence of lenticularity of the sands.

A. H. N.

1308.* Consistent Records Vital to Efficient Pumping. F. B. Taylor. *Oil Wkly*, 19.6.39, **94** (2), 16–22.—Properly kept records form means for an unbiased analysis of conditions and troubles that often lead to corrective measures. With this theme as his thesis, the author discusses the value of records, and urges the keeping of complete yet concise records which not only tell the facts about changes and alterations, but also record the circumstances and causes which necessitated the changeover. Sample sheets and forms are presented for well-repair cost record, well-repair job sheet, daily well-trouble report, well-inventory report, summary of well expenses, service record-data sheet, equipment and operating report, shop reports, sucker-rod record, flat-belt service record, and " V "-belt service record. A. H. N.

1309.* Aplication of Radial Flow Calculations to Gas Recycling Problems. L. S. Reid. *Oil Wkly*, 26.6.39, **94** (3), 17.—Because injection wells are considered "necessary evils," the ratio of producing to injection wells is kept from 2 to 4 with the latter working at full capacity. Muskat's analysis, which shows how the productivity of a formation can be calculated through the analysis of the rate of the pressure rise in a well which has been shut in for a certain period of time, is taken as a basis for this study. The hypothesis that the production rate is proportional to difference between the static and flowing bottom-hole pressure is accepted, and a formula is obtained defining this relationship in terms of pressures (P and P_e), thickness (b), and permeability (k) of the rock, and viscosity (μ) of fluid and the ratio of the radius of the well (r_w) and an external radius (r_e), where the pressure is equal to the reservoir pressure (P_e). The formula is—for radial flow

$$Q = \left[\left(\frac{2\pi kb}{\mu}\right)\Big/\log_e\left(\frac{V_e}{V_w}\right)\right](P_e - P)$$

where P is the bottom-hole pressure of the flowing well.

The method of measuring bottom-hole pressure advocated in a gas well is to measure the casing-head pressure and calculate the bottom-hole pressure by adding the hydrostatic head of fluid filling the well. Thus a column of gas in an 8000-ft.-deep well under a surface pressure of 3,500 lb./sq. in. (gauge) and 125° F. with a molecular weight of 25 and a deviation factor of 0·8 is taken as an example and the bottom-hole pressure is calculated at 4625 lb./sq. in. absolute by utilizing the formula $\int Vdp = \int \frac{RT}{P}\,dp =$ 8000 ft. of fluid. If the well was flowing so that surface pressure was 3000 lb./sq. in. the bottom-hole pressure would be 3970 lb./sq. in. absolute or a drop of only 155 lb./sq. in. in the hydrostatic head. In view of the relatively small drop in the hydrostatic head, it is believed that build-up curves can be used satisfactorily in estimating the pressure required for the injection of gas back into the reservoir.

Examples are also worked out to calculate the permeability of a sandstone and a limestone formation from a graph of pressure rise in casing for a well in the formation, using Muskat's formula for radial flow of gas in porous media.

Pressure differential between input and output wells and well patterns are discussed. The point of injection, with regard to the flanks or top of structure, is important. Consensus of opinion tends to support the top of the structure, as there is less danger of the gas being near the edge-water. The practice of gas recycling has been so recently developed that the superiority of one spacing pattern over another has not been established by actual operation, as has been the case in water-flooding.
A. H. N.

1310.* Light Pumping Foundations Frequent Sources of Trouble. F. B. Taylor. *Oil Wkly*, 26.6.39, **94** (3), 21.—Foundations for light-weight field equipment are too often the cause of trouble and lost production. Difficulties may reassert themselves several times. Maintenance of foundation is a field expense always kept as low as possible, and hence repairs are too frequently inadequate.

The solution is not in complete and costly repairs, but in building a foundation in the first place to satisfy both conditions of operation and of equipment.

The paper deals exclusively with light foundations, and treats the problem under two major types of foundations :

(1) foundation for jacks ;
(2) foundations for single power pumpers of small power.

Massive foundations used with unit pumpers or lifts exceeding 6 or 7 h.p. are explicitly excluded from the discussion.

Newer methods of foundation design take fuller account than older empirical methods of the direction of stress and pull, and lay the foundations with as much area exposed as bearing surface in the direction of power as is possible. A. H. N.

1311.* East Texas Expected to Go On Pump at Increased Rate. B. Mills. *Oil Wkly*, 17.7.39, **94** (6), 34.—The East Texas field at the present time is still predominantly a flowing field, even after producing 1,370,000,000 brl. of oil in 8¾ years, an amount that exceeds the life time-quantity recovered from any field with the exception of Baku. Eventually, however, it will undoubtedly become a very extensive pumping area. The question whether pumping equipment will have to be installed at a faster rate in the future than in the past is a matter of prime importance to operators in the field. Indications show that it will, as the flowing life of the field has already been much longer than was predicted at the time of its discovery. A. H. N.

1312.* Ramsey Proving Value of Unit Development of Small Pool. B. Altman. *Oil Wkly*, 31.7.39, **94** (8), 41.—An outline is given of the development of the Ramsay Pool, Payne County, Oklahoma. The area is an example of the benefits attending unit operations. Structurally the field is a small dome with deep dip in all directions. The structure was found by seismograph explorations, and the discovery well was brought in in January 1938. Production is from the Wilcox sand at 4730—4800 feet. Bottom-hole pressure of the discovery well was 2038 lb. ; so far the decline in pressure has been negligible. No free gas exists in the reservoir. A brief description of the drilling and completing methods employed is given. A. H. N.

1313.* Wasson-Bennett Merging to Make Largest, Most Active Field in Permian Basin Area. H. H. King. *Oil Wkly*, 7.8.39, **94** (9), 20.—The development of, and current operations in the Wasson–Bennet district of Gaines–Yoakum counties, West Texas, are given, with an outline of the drilling and completing practices employed there. The Wasson field has a proved area of 34,000 acres, and the Bennet field one of 3000 acres. Ultimate crude recovery from the known producing horizon in the district will probably exceed 250,000,000 brl. Regulations of the Texas Railroad Commission permit a maximum gas–oil ratio at Bennett of 2000 cu. ft. of gas per barrel of oil, whilst at Wasson the top ratio is 5000 cu. ft. of gas per barrel. A. H. N.

1314.* Sound Economics Must Be Teamed with Science in Future Course of Oil. F. Phillips. *Oil Wkly*, 14.8.39, **94** (10), 13.—The author attacks the uneconomic practices employed in the recovery of petroleum in the United States, more especially the excessive and competitive drilling which has merely resulted in smaller financial return to the industry. He pleads strongly for the adoption of unitization in the exploitation of oil-fields and for wider well spacing. Examples are cited of the waste attendant upon over-drilling of oil-pools and of the benefits that have accrued in other areas where producers have followed a unit plan of development. Mr. Phillips thinks that unitization, wider well spacing, and similar ideas for recovering ultimately the greatest amount of oil at the lowest possible cost will soon be universally recognized and applied by the American petroleum industry. But the necessity for unitization is urgent. A. H. N.

1315.* Texas' First Water-Flooding Project Big Success. J. C. Albright. *Oil Wkly*, 21.8.39, **94** (11), 36.—The history of the water-flooding projects in Texas is given from early days up to the present. Earlier neglect of scientific principles are discussed with their resultant troubles. The methods of conditioning and injecting the water to-day are given in detail, with a plan of the water-treatment plant for flooding oil sands on a typical field. The ratio of produced oil to injected water works out at 1 : 11. It appears that the flood has progressed to a point where the water has been distributed evenly over the lower portions of two sands. A. H. N.

1316.* Human Element in Pumping Efficiencies. F. B. Taylor. *Oil Wkly*, 28.8.39, **94** (12), 29.—A detailed discussion of the various human factors entering into the efficiency of pumping wells is made according to the thesis of the author, "the engineer, the manufacturer, and the pumper must work together, or the best of equipment will be of little value." A. H. N.

1317.* Reaction to Draw-Down Methods of Potential Determination in Kansas Both Favourable and Adverse. W. H. Strang. *Oil Wkly*, 5.6.39, **93** (13), 31.—"Draw-down" refers to the reduction in height of the fluid column standing in the annular space. The footage difference in height of the fluid-level under static conditions and fluid-level height at a given rate of production is the draw-down for the rate of production. Productivity index is defined as production rate in brl./day per foot of draw-down, and when this factor is multiplied by the static fluid-level height, it will give a potential for the well.

Two types of instruments are described for measuring fluid levels by means of reflected pressure waves from the fluid level. Knowing the speed of the wave, and determining the time it takes to travel to or from the fluid-level, the height of the level may be assessed. When the wave travels from one gas into a denser one, the speed of the wave falls, and hence complications arise in wells which have stratified conditions of gas.

Other methods of tests are described, certain of them with particular reference to wells which do not produce free gas. Difficulties peculiar to certain wells, such as absence of markers in the well, in the form of tubing-catchers or liner-tops, are discussed.

It is shown that draw-down is proportional to rate of production by means of graphs.

The two other systems of determining potential, by pressure bomb method and 8-hr. production test, are described and discussed. A controversy rages around which method is the most accurate and fair. A. H. N.

1318.* Production Problems Contended With at Sac and Fox Pool. P. Reed. *Oil Gas. J.*, 1.6.39, **38** (3), 32.—The two chief troubles encountered in these areas are flooding on the surface and wax deposits. Detailed considerations are paid to the first problem, and provisions are made to work on raised levels in times of floods. To remove wax, lead lines are taken up every 90 days and brought to a boiler, where they are steamed out. Scrapers are sufficient in hot weather.

The history of the field is given. A. H. N.

1319.* Oklahoma City Field Enters Secondary Recovery Stage. H. F. Simons. *Oil Gas J.*, 6.7.39, **38** (8), 28.—Oklahoma City field is facing a prospective revival through the introduction of secondary recovery methods. In this paper, the first of a series of two articles, the author summarizes the present conditions and possible future of the Oklahoma City field.

A unique method of obtaining potential was instituted recently. An operators' committee, composed of engineers familiar with the field, arrived at estimates of what the potential would be if tested under periods of varying lengths. The estimates were well received, and it was decided to refine the work further and to arrive at a potential by sound engineering methods that would be agreeable to all parties.

The working of the scheme is summarized. A. H. N.

1320.* Acidization of Gas Wells in the Texas Panhandle. D. H. Stormont. *Oil Gas J.*, 6.7.39, **38** (8), 33.—The first gas well acidized in the Panhandle field was in 1937, and much progress has been made since then. Use of acid is now adopted as a standard completion practice by many operators in this field. Also many operators are treating some 10- or 12-year-old wells.

Treatment of gas wells, in general, is along the lines used in oil-well acidization, with the exception of some differences in the equipment used and the method employed. Stage treatment is now employed.

In acidizing oil wells oil is used as the loading to force the acid into the formation. In treating gas wells this is not possible, as there is a likelihood of the oil decreasing the permeability of the pay sand. Thus either compressed gas or water is used, with the former being more widely used since it is cheaper, more readily available, and has been found to give better results. Also, water requires swabbing, which entails, generally, moving a swabbing machine to the well, and thus increasing the treating cost.

Use of addition agents designed to reduce the surface tension of the acid has greatly increased the efficiency of the methods used.

A detailed study of the many factors peculiar to the field and the work is given.

A. H. N.

1321.* Theories and Practices in Modern Oil-Well Shooting. R. B. Bossler. *Oil Gas J.*, 20.7.39, **38** (10), 34.—Considerable speculation as to the effects produced by the explosion still exist, although the purpose and effects of shooting oil-wells are well known. The author attempts to bring together some of the latest work on the subject, including his own field observations and deductions. The assumption that the creation of a cavity was the main object of shooting is now abandoned. Such assumptions give insufficient explanation of the results obtained. A. H. N.

1322.* World Crude Production. H. S. Norman. *Oil Gas J.*, 27.7.39, **38** (11), 56.—For the first six months of 1939 petroleum output of the world established a new half-yearly peak figure. Production amounted to over 1010 million brl., or nearly 33 million brl. above the output for the corresponding period of 1938. A. H. N.

1323.* Crude-Oil Reserves Increased in First Six Months. H. F. Simons. *Oil Gas J.*, 27.7.39, **38** (11), 62.—This is the *Oil and Gas Journal* half-yearly review of operations and developments in the United States petroleum industry. Discussing crude reserves, it is stated that new oil discovered in the first six months of 1939 amounted to roughly 1184 million brl. New pools promise nearly 240 million brl., and extensions to older fields account for the balance. The most important discovery of the period was the Magnolia field, Columbia County, Arkansas, which is credited with an ultimate recovery of 150 million brl. Deducting the production of 616½ million brl., the net gain in American crude-oil reserves during the first half of this year was 567½ million brl. A. H. N.

1324.* Major Areas Developed as Rice Country Pools are Extended. D. Dalrymple. *Oil Gas J.*, 3.8.39, **38** (12), 32.—One of the larger oil areas of the United States is the Rice County field, Kansas. It has a potential yield of nearly 1 million brl. of oil daily, and this potential promises to increase. New discoveries are tending to offset the natural decline in older pools. The early history of the field is outlined from 1924 up to recent developments. A. H. N.

1325.* East White Point Field, Texas. Anon. *Oil Gas J.*, 3.8.39, **38** (12), 37.—The East White Point field, San Patricio County, South-west Texas, is discussed thoroughly. The discovery well was completed in February 1938. There are 95 producing wells in the field. Production is obtained at about 5650 ft. All wells flow naturally: working pressures at the surface range from 500 to 1000 lb. on tubing and from 750 to 1200 lb. on casing. Bottom-hole pressure is 2500 lb. Gas/oil ratios in the field range from 500 to 600 cu. ft. of gas per barrel of oil. East White Point crude has a gravity of 39 degrees A.P.I., a gasoline content of 38%, and a gas oil content of 41·2%. Other data of interest are given. A. H. N.

1326.* Major Companies Control Bulk of Illinois Production. H. F. Simons. *Oil Gas J.*, 10.8.39, **38** (13), 31.—Salem, Louden, and Clay City constitute the main production areas in Illinois. They account for 199,000 brl. daily, or roughly eight-ninths of Illinois' total oil output at the time when the State was producing 225,000 brl. a day. Two-thirds of Illinois production is controlled by the major oil companies. The present leader in production, number of wells, and potential output is the Texas Corporation, and the second and third are the Carter Oil Co. and the Pure Oil Co., respectively. On 1st July last these three concerns alone had 1728 wells and a daily production of 109,900 brl. in Illinois. While the aggregate output of the larger companies is higher than that of the independent interests, the per-well yield of the major companies is less than that of the independents. For instance, the average-per-well yield of the major interests in the Salem field at the end of June was 96 brl. per day, whereas the average of the small companies was 300 brl. per day. A. H. N.

1327.* Magnolia Field Columbia County, South Arkansas. Anon. *Oil Gas J.*, 17.8.39, **38** (14), 44.—A general account is given of the Magnolia field, Columbia County, South Arkansas. The field was discovered in February 1938. At present there are 50 producing wells in the field and the proven acreage amounts to 1750 acres. The original reservoir pressure was 3575 lb.; present pressure is 3358 lb. An analysis of the Magnolia crude is given. The field has an estimated ultimate recovery of 150 million brl. of crude. A. H. N.

1328.* Manifold Hookup on a Coastal Louisiana Production Property. N. Williams. *Oil Gas J.*, 31.8.39, **38** (16), 36.—A simplified, unusually flexible, and low-cost manifold has been developed by a Gulf Coast, Louisiana, oil producer and is described with respect to its construction and operation. A schematic chart is given. A. H. N.

1329.* Production Problems Loom for Shreveport Field. G. Weber. *Oil Gas J.*, 31.8.39, **38** (16), 47.—The Shreveport Field of Caddo Parish, North Louisiana, has entered its second year of development with a total of 48 wells and a daily average production in excess of 7000 brl. Although the pressure-production relationship in the field is discouraging at this time, conditions would have been much worse had not operators and State conservation engineers adopted sound conservation measures in developing it.

Tests in numerous wells show that the degree of permeability is much greater vertically than horizontally. This factor tends to retard lateral migration of oil to the well, favouring instead, vertical movement and eventual coning of the underlying water. Thus, it is indicated that in the future the field may enter a stage of more gradual pressure decline if production is controlled to prevent water intrusion at too early a date. A. H. N.

1330.* Production Patents. H. U. Garrett. U.S.P. 2,156,429, 2.5.39. Appl. 26.4.37. Bottom-hole control choke, consisting of a perforated casing and a tubing carrying two packers to be set above and below the gas sand, and thus form a pressure chamber.

C. C. Taylor. U.S.P. 2,156,709, 2.5.39. Appl. 20.4.36. A well cleaner and tester.

K. T. Penick. U.S.P. 2,157,496, 9.5.39. Appl. 16.12.35. Combined tubing head and braden head.

A. N. Porter. U.S.P. 2,157,628, 9.5.39. Appl. 11.1.37. An oil-well pumping rig.

J. P. Jackson. U.S.P. 2,157,704, 9.5.39. Appl. 9.10.35. A deep-well pump with two cylinders and pistons working simultaneously.

C. I. Leibensperger and F. H. Thorpe. U.S.P. 2,157,713, 9.5.39. Appl. 1.6.37. Process of manufacturing sucker-rod couplings and couplings so made.

W. L. Church. U.S.P. 2,157,729, 9.5.39. Appl. 17.5.37. Apparatus for bringing a well into production by gun perforation.

R. A. Mueller. U.S.P. 2,157,964, 9.5.39. Appl. 16.2.34. Tubing hanger in a casing-head provided with a stuffing-box so that the tubing may be reciprocated during the washing of the well.

H. E. Aulman. U.S.P. 2,158,393, 16.5.39. Appl. 2.4.38. A water-well casing.

L. Bowen. U.S.P. 2,158,569, 16.5.39. Appl. 24.5.38. A formation tester comprising a tail tube and a packer, the tube having a fine perforated test conduit and a pressure recorder responsive to pressures within the perforate extension and a relatively coarse-perforate member below the first member and out of communication therewith, and a pressure recorder responsive to pressures within said coarse-perforate member. A. H. N.

Transport and Storage

1331.* A Field Balance for Weighing Pipe to Determine Rate of Corrosion. T. H. Marshall and D. Chads. *Petr. Engr*, June 1939, **10** (9), 67–74.—It is pointed out that corrosion investigations carried out in the laboratory are not satisfactory, as field conditions cannot be reproduced. Corrosion experiments under field conditions have been made possible by the construction of a suitable balance. This balance consists of a supporting tripod, beam assembly, sample suspension sling, and a suspension device for the weights, and has a beam ratio of 5 : 1. It is easily portable, and strong enough to weigh a 10-ft. section of standard pipe weighing 30,000 gm. Tests have shown that the balance gives results to seven significant figures, as compared with six normally obtained with an analytical balance. A detailed description of the construction of the balance is supplemented by drawings. Results are vitiated by variation of air humidity. R. J. E.

1332.* Developments in Procedure for Installing Cathodic Protection to Pipe Lines. W. F. Rogers. *Petr. Engr*, Mid-Year 1939, **10** (10), 112–116.—The procedure adopted in the installation of cathodic protection is outlined. The recording of repairs to pipe-lines is recommended and a typical chart of cumulative rust-hole data is shown. From these data the future history of the pipe is predictable, so that the anticipated cost of repair can be compared with the cost of re-coating and applying cathodic protection. Alternatively, the application of cathodic protection alone may be considered.

Some field work is necessary to estimate the cost of cathodic protection. For a well-coated pipe a minimum potential of 0·80 volt is considered to give adequate protection. Measurements are made to determine the current required to maintain this potential, consideration being given to the effect of seasonal changes, and moisture content on the pipe, to soil potential and to the potential developed by the deposition of hydrogen on the pipe. The spread of current on the pipe is determined and data are obtained to show the length of pipe protected. Consideration is given to the spacing, composition, and resistance of the ground bed. From the data obtained the cost of cathodic protection is calculated. The use of engine-driven generators is recommended as the cheapest method of providing cathodic protection. R. J. E.

1333.* Method of Determining whether Electrical Protection should be Applied to a Pipe-line or the Line Reconditioned. D. B. Good. *Petr. Engr.*, July 1939, **10** (11), 29–30.—The application of cathodic protection is considered in relation to two different cases. First, where corrosion has already occurred, and secondly, where no corrosion is present but where damage would prove expensive and difficult to repair. In the first case it is assumed that a record of corrosion and maintenance costs of the pipe-line for a number of years is available. Preliminary resistivity measurements are made to determine the point at which the protecting unit is needed. Curves relating pipe to ground potential and current are then obtained and the amount of pipe protected by various currents is deduced. The voltage required is obtained from previous knowledge of the test ground or by soil resistivity measurements. Consideration is given to variations of pipe to ground potential with the seasons. From the data obtained the type of equipment necessary can be selected, the power consumption deduced, and the cost of electrical protection compared with the cost of reconditioning or maintenance. Where preliminary tests show that more than one unit is necessary, it is found that the length of pipe protected per unit is considerably greater than in a single unit operation, and simultaneous tests are made for comparing the cost of operation and installation with reconditioning or maintenance costs. In cases where only short lengths of pipe are affected, electrical protection shows to less advantage in comparison with reconditioning.

In the second case, measurement may indicate current losses and the adoption of electrical protection to be justified, although no corrosion has been observed. Moreover, even if no current losses are detected, high cost of reconditioning may justify cathodic protection as a precautionary measure. R. J. E.

Crude Petroleum.

1334.* Kulsara Crude. I. V. Vysotski. *Neft. Khoz.*, June 1939, **20** (6), 46–49.—Kulsara crude (from well No. 10) is a light, low-asphaltic crude oil with a high wax content. It is of paraffinic base and very rich in gasoline-ligroine fractions, 68·8% distilling below 300° C. (Engler).

The gasoline-ligroine fractions can be classified as follows: (*a*) aviation gasoline fractions, 29·2 and 39·3% distilling between I.B.P. and 130, or 160° C., respectively; (*b*) motor-car gasolines, 44·3 and 48·8% distilling between I.B.P. and 180° or 200° C. As the gasoline fractions, however, have a distinct paraffinic character, it is unlikely that high octane fuels could be obtained in the usual way.

23·5% of the crude distils between 200° and 300° C., and can be utilised for producing various grades of illuminating kerosine. Residual oil (28%) remaining after distilling off the light fractions (up to 300° C.) has an extremely low specific gravity ($d_4^{20} = 0{\cdot}8606$) and a high pour point (+22° C.). After refining and dewaxing various high-grade residual oils can be obtained, including bright stock and aviation lubricating oils.

L. R.

1335.* Unusual Crude from Old Ocean Field, Brazoria County, Texas. Anon. *Petr. Engr*, July 1939, **10** (11), 62.—On analysis the crude was found to be lighter in colour than No. 1 N.P.A., low in sulphur, and yielded 71% of 200° C. E.P. gasoline and 14% of kerosine distillate, the remainder being graded as gas-oil. R. J. E.

Gas.

1336.* Progress in the Manufacture of Butane. J. W. Vaiden and F. E. Fisher. *Petr. Engr*, Mid-year **10** (10), 128–129.—Increased demand for propane and butane as a domestic and industrial fuel and the requirements of polymerization and synthetic processes have stimulated progress in their manufacture. A trend to higher quality has stimulated the construction of new plant for the efficient production of a high-purity product. Methods used to obtain the desired results are one or more of the following: high-pressure compression and absorption, refrigeration and vapour rectification. Changes in existing plant to give the desired results consist of higher pressures, increase in rate of absorption, lower oil temperature, refrigeration under high pressure, vapour rectification at low temperature, and liquid rectification at high pressure. The utilization of propane as a refrigerant, and also the construction of two modern plants, are described. Attention has been paid not only to processing plant, but also to the provision of efficient equipment for safe handling and storage of the hydrocarbons.

The increased importance of repressuring has focussed attention on the recovery of hydrocarbons from the gas before it is returned to the oil sands. R. J. E.

Cracking.

1337.* Houdry Process of Catalytic Cracking. M. Houdry. *Ass. franc. Tech. Petrol. Bull.*, 1.5.39 (49), 5–14.—A description is given, with flow-sheet and properties of the products, for a catalytic cracking process operating on typical crudes.

The crude first undergoes fractionation into straight-run spirit naphtha for reforming, and a fraction which, after separation of tar, forms the charge for the catalytic cracking unit. This operates under atmospheric pressure and at a moderate temperature. The cracked products are admitted to a fractionating column, the light products being separated into spirit and gas for reforming and polymerization. The high-boiling product is an excellent gas-oil, which may be cracked further if necessary.

When cracking crude or residual stock a vaporizer is interposed between the initial fractionating column and the catalyst chambers, the tar separator being by-passed.

A comparison is given of results on Lagunillas crude using two different vaporizers.

For Iraq crude a 61·5% conversion into 74·7 octane gasoline when operating on crude, and 76·5% on cracking stock, is obtained.

Using East Texas crude a 74·2% conversion into 74·4 octane gasoline is obtained when operating on crude, and an 80% conversion into 74·7 octane gasoline on cracking stock. The process may readily be adapted to yield aviation spirit base of 79·8 octane number, having low sulphur and gum content.

Units are being constructed in France to come into operation in June 1939 on the basis of an annual output of 3½ million tons. W. S. S.

Refining and Refinery Plant.

1338.* Application of Automatic Controls. A. J. Foley. *Refiner*, 1939, **18** (8), 301.—Maintaining that the application of an automatic control instrument is of far greater importance than the instrument itself, the author cites several cases in the petroleum industry where the application of control had not the desired effect. These cases include thermal control of throughput on a combination unit, liquid level control of an absorber on a gasoline plant, liquid level control on the residue stream of a vacuum tower, and a pressure reducing regulator on a line between two distant gas-storage tanks. G. R. N.

1339.* Modern Instrumentation. D. J. Bergman. Refiner, 1939, **18** (8), 304.—Stability or smoothness of operation is the most desirable feature in automatic control systems for distillation, cracking, and polymerization plants. As a rule, flow control of feed is one of the major factors in obtaining process stability. In the simplest type of distillation unit automatic control is provided by flow control on the crude-oil charge, temperature control on the heater transfer line, control of the gasoline quality by means of tower top temperature with a valve in the line from the reflux receiver to the top bubble tray, and liquid level control at the bottom of the tower. In the conventional two-coil Dubbs unit fixed flow to the heaters is the heart of the control system. Transfer temperature is regulated by hand control using pressure-balanced valves in the gas lines to each heater. A pressure-control valve maintains constant pressure on the reaction chamber and a liquid level controller releases residuum from the bottom of the flash-chamber. Gasoline is controlled as in a distillation unit. Receiver gas is released by a pressure controller and metered, while receiver distillate passes via a flow controller to the stabilizer. The minimum instrumentation for a small U.O.P. catalytic polymerization unit is also described. It is essential that the maximum throttling range of the instruments be used in order to avoid hunting. Allowances must also be made for the tendencies of the equipment towards self-regulation. G. R. N.

1340.* Cooling Condenser Water with Spray Ponds. J. D. Watson. *Fuel Econ.*, 1939, **15**, 178–180.—When sufficient space is available around a works there is much to be said for handling the condenser water with a spray system. Normally the cost of installing such a system is less than for a natural-draught tower. Its cooling range is lower, but pumping costs are less. Providing the installation is correct, spray cooling can be as efficient as tower cooling.

Frequently the spray system has been condemned, when actually the installation of the system has been at fault. The ideal arrangement of the system and the types and setting of nozzles are outlined. T. C. G. T.

1341. The Cooling of Condenser Water. J. D. Watson. *Fuel Econ.*, 1939, **15**, 207–210.—A review of cooling towers, their types and construction, the Davenport, Foster Wheeler, and the louvred type manufactured by the Premier Cooler & Engineering Co. are described and illustrated with photographs and diagrams. T. C. G. T.

1342.* Flow Resistance of Viscous Fluids in Pipes. A. Skryabin. *Neft. Khoz.*, April–May, 1939, **20** (4–5), 67–68.—An equation is evolved for the flow resistance of two-phase liquids under laminary flow and non-isothermic conditions :

$$\mathrm{H} = \left(\frac{128Q\eta_0 l}{\pi d^4 g 10^4} + \frac{16l\Theta_0}{3gd10^4}\right)\left(\frac{l^{nC} - 1}{nC}\right)$$

where H means pressure drop in m. water ; Q throughput in cu.m./sec. ; l pipe-length in m. ; d diameter in m. ; g acceleration of gravity in m./sec.^{2} ; C heat capacity ; η_0 viscosity in poises at the beginning of the pipe ; Θ_0 initial breakaway tension in dynes/sq. cm. ; and n a constant $= 3{\cdot}5$.

For highly viscous liquids without initial breakaway tension (*e.g.*, residual oil) the formula is simplified :

$$H = \frac{128Q\eta_0 l}{\pi d^4 g 10^4}\left(\frac{l^{nC} - 1}{nC}\right)$$

L. R.

1343.* Analysis of Power Requirements in Refinery Operations. W. H. Stueve. *Petr. Engr*, June 1939, **10** (9), 120–128.—A comprehensive survey of the power requirements of U.S. oil refineries has been made from the point of view of the potentiality of oil-processing as a market for electric power sold by electric utility companies. Refineries are classified into : (*a*) skimming—gasoline recovery by distillation only, (*b*) skimming and cracking, and (*c*) complete, recovery of lubricating oil as well as skimming and cracking, and a detailed analysis of the power requirements of each class is given. It is concluded that a modern refinery uses 0·42 kw-hr. more electric power for every barrel of oil processed than an older type. A classification of refineries on a geographical basis shows the effect of location on the fuel used, *e.g.*, oil predominates in East Coast, coal in Appalachian, and natural gas in Arkansas refineries. The effect of new developments in the oil industry is illustrated by the possibility of the replacement of energy from refinery gases by purchased electric power as a result of the potential increase in the utilization of the gases in polymerization processes.

R. J. E.

1344.* Developments and Trends in the Natural Gasoline Industry. J. W. Vaiden and F. E. Fisher. *Petr. Engr*, (Mid-Year 1939), **10** (10), 44–48.—Developments of the natural gasoline industry are outlined and the effect on this industry of recent progress in the rest of the petroleum industry is discussed. Advancement of plant design is exemplified by the erection of refrigeration plants which are claimed to be more economic than conventional absorption or compression–absorption plants. Refrigeration has made possible efficient recovery of propane and butane at nominal pressure. Freezing difficulties experienced in refrigeration have resulted in intensive study of hydrate formation and the development of dehydrating processes. Progress has been made in the study of de-sulphurization to improve lead susceptibility. Better recovery of lower hydrocarbons by low-molecular-weight oils has resulted in the use of lighter oils for absorption. The application of repressuring has been attended by the development of high-pressure absorption, and operation at 1500 lb. has been successful.

The progress made by the refiner in the better utilization of his raw material tends to decrease the demand for natural gasoline, but increase in the octane rating of marketed gasoline makes possible an increased demand, owing to the good anti-knock value and lead susceptibility of natural gasoline. The synthetic processes of the refiner are open to the natural-gasoline manufacturer, and the adoption of dehydrogenation, polymerization, or alkylation would enable him to convert a low-price material into a high anti-knock premium product. R. J. E.

1345.* Heater for Treating Cut Oil. J. C. Albright. *Petr. Engr*, July 1939, **10** (11), 64.—The production of cut oil from some wells in Oklahoma has resulted in the installation of small portable gas-fired heaters. These heaters are installed in the lead lines from the wells between the tubing head and the flow tanks, and may be set to deliver a constant temperature. A detailed description of the construction of a heater is accompanied by an illustration. R. J. E.

1346.* Problems Arising from the Presence of Salts in Crude Oils. J. V. Canessa and M. Fernandez Romero. *Bol. Inform. Petroleras*, June 1939, XVI (178), 12–16.—The percentage of mineral salts present in crude petroleums varies greatly in different fields all over the world and also locally at different depths of the wells. The highest percentages are to be found in the Argentine, where an average taken from a number of samples shows as much as 2,226·5 gm. of sodium chloride per cubic metre. The article discusses the question generally and points out the serious damage which is caused to stills, fractionating stills, cracking towers, and condensers. The salts occur in the form of solution in water and as crystals surrounded by a skin of paraffin or asphalt.

The methods of dealing with these salts are indicated broadly, but another article in the same number treats the matter in fuller detail. There are four groups of treatment by physical processes and six different chemical methods of attacking this very serious trouble. References are given to recent literature on the subject. H. I. L.

1347.* Industrial Processes for Removing Salts from Crude Oils. B. Rikles and J. Stowasser. *Bol. Inform. Petroleras* XVI (178), 17–30.—This article contains a detailed account of the various processes employed which are most appropriate to the varying condition of the petroleums. These processes which have proved efficient, are

1. Electrical processes.
2. Decanting with application of heat and pressure.
3. Chemical processes.
4. Contact with solid materials followed by filtration.
5. Centrifuging.
6. Combination of electrical and chemical process.

An ample bibliography is added for reference. H. I. L.

1348.* Treatment of Petrols and its Chemical Aspects. A. F. Sainz. *Bol. Inform. Petroleras* June 1939 XVI (178), 31–36.—The article deals with treatment, (*a*) by absorbents, and (*b*) by chemicals. Various processes are detailed, and special mention is made of cracking which is accompanied by the production of sulphides, resins, etc., which have to be eliminated. H. I. L.

1349.* Y.P.F. Refineries—Improvements Installed in Record Time. Anon. *Bol. Inform. Petroleras*, June, 1939, XVI (178), 3–11.—Illustrated description of displacement of a cracking tower at a Y.P.F. refinery to a site at some distance and the installation of a new tower in its place. It is shown how the work was planned so as to secure minimum interruption in the working of the refinery. H. I. L.

Chemistry and Physics of Petroleum.

1350.* Thermal Dehydration of Ethane to Ethylene. L. Potolovskii and A. Atal 'Yan. *Petr. Engr*, July 1939, **10** (11), 40–48.—A comprehensive study of the pyrolysis of ethane over a temperature range of 675–900° C. with reaction periods ranging from 0·01 to 13 seconds is described. The investigation was directed towards determining the optimum conditions for the production of ethylene. At 675–850° C. for 0·3 to 0·6 sec. ethylene, methane, hydrogen, and hydrocarbons higher than C. 4 are formed. At higher temperatures acetylene is also produced. The maximum yield of ethylene from the ethane decomposed was obtained at 675–725° C., but a relatively small proportion of the ethane was decomposed. Above 850° C. the yield of methane, hydrogen, acetylene, and higher hydrocarbons increases rapidly. Pyrolysis at 875–900° C. for 0·05 sec. resulted in the decomposition of 85% of the ethane giving a 62% yield of ethylene. At 700–750° C. the greatest yield of ethylene was 40–45% in a reaction time of 2·9–7·0 secs. Increase of the reaction time reduced the amount of ethylene and increased the methane. It is concluded that the best conditions for the pyrolysis of ethane are preheating to 700° C. followed by instantaneous high-temperature pyrolysis in a tubular furnace of special steel capable of withstanding prolonged heating at high temperatures. R. J. E.

1351. Theory of Absolute Reaction Rates and The Polymerization of Ethylene. F. P. Jahn. *J. Amer. chem. Soc.*, 1939, **61**, 798–800.—This is a theoretical paper in which the activated complex method used by Eyring is employed to calculate the rate of homogeneous thermal polymerization of ethylene: by making simple assumptions concerning the entropy of activation rough agreement with experiment is obtained. R. D. S.

1352. Critical Constants of Ethane. J. A. Beattie, Gouq-Jen Su, and G. L. Simard. *J. Amer. chem. Soc.*, 1939, **61**, 924–925.—The authors have investigated the critical constants of ethane by the compressibility method and found them to be :—

$t_c = 32{\cdot}27 \pm 0{\cdot}01°$ (Int.),
$p_c = 48{\cdot}20 \pm 0{\cdot}02$ normal atmospheres,
$v_c = 0{\cdot}148$ litre per mole (4·93 cc. per gram),
$d_c = 6{\cdot}76$ moles per litre (0·203 gram per cc.)

The uncertainty in the critical volume and density is 1%. R. D. S.

1353. Compressibility of Gaseous Ethane in the High-Density Region. J. A. Beattie, Gouq-Jen Su, and G. L. Simard. *J. Amer. chem. Soc.*, 1939, **61**, 926–927.—The authors give compressibility measurements of ethane from 50 to 275° and from 5 to 10 moles per litre. R. D. S.

1354. Reactions of 3-Hexene. II. Condensations with Aromatic Hydrocarbons and Phenols. L. Spiegler and J. M. Tinker. *J. Amer. chem. Soc.*, 1939, **61**, 1002–1004.—3-Hexene has been condensed with hydrocarbons and phenols using a series of catalysers such as sulphuric acid, anhydrous hydrogen fluoride, dihydroxylfluoboric acid ($H_3BO_2F_2$), perchloric acid, zinc chloride, and aluminium chloride.

1, 2, or more hexyl groups were introduced into the aromatic nuclei to obtain the corresponding secondary hexyl derivatives. T. C. G. T.

1355. Hydrofluoric Acid as a Condensing Agent. II. Nuclear Alkylations in the Presence of Hydrofluoric Acid. W. S. Calcott, J. M. Tinker, and V. Weinmayr. *J. Amer. chem. Soc.*, 1939, **61**, 1010–1015.—It has been shown that hydrofluoric acid is an effective acid condensing agent for the preparation of nuclear alkylated isocyclic compounds.

It was possible to introduce into isocyclic compounds alkyl groups containing as few as two carbon atoms, but the best results were achieved when alkyl radicals containing three or more carbon atoms were used.

Olefines or compounds which under the conditions of reaction would be expected to react like olefines, such as alcohols, esters, ethers, or halides, were employed as alkylating agents. T. C. G. T.

1356. The Nitrogen Compounds in Petroleum Distillates. XIV. Isolation of 2 : 4-Dimethyl-8-Ethylquinoline from the Kerosene Distillate of Californian Petroleum. W. N. Axe. *J. Amer. chem. Soc.*, 1939, **61**, 1017–1019.—The isolation from a complex mixture of petroleum bases, of the hitherto unknown compound 2 : 4-dimethyl-8-ethylquinoline, is described. The structure of this compound has been established by oxidation to 2 : 4-dimethylquinoline-8-carboxylic acid and by synthesis of the compound. T. C. G. T.

1357. Simultaneous Dehydrogenation-Hydrogenation of *Cyclo*hexene in the Presence of Nickel. B. B. Corson and V. N. Ipatieff. *J. Amer. chem. Soc.*, 1939, **61**, 1056–1057.—In the presence of nickel, both at superatmospheric and at ordinary pressure, *cyclo*hexene is converted almost quantitatively into a mixture of *cyclo*hexane (68% by weight) and benzene (32% by weight).

This is in contradiction of earlier workers, whose results indicated that nickel was not such an effective catalyst. T. C. G. T.

1358. Isomerization of Akyl*cyclo*pentanes. H. Pines and V. N. Ipatieff. *J. Amer. chem. Soc.*, 1939, **61**, 1076–1077.—Alkylated *cyclo*pentanes have been isomerized to yield methylated *cyclo*hexanes. Thus at 50° C. in the presence of aluminium chloride ethyl*cyclo*pentane yielded methyl*cyclo*hexane; propyl- and isopropyl-*cyclo*pentane gave 1 : 3-dimethyl*cyclo*hexane, whilst *n.*-butyl-, *s.*-butyl-, and *t.*-butyl*cyclo*pentane yielded 1 : 3 : 5-trimethyl*cyclo*hexane.

The yield of isomerized product in most cases exceeded 80%. T. C. G. T.

1359. The Formation of Intermediate Compounds in Hydrocarbon Syntheses by the Friedel and Craft Reaction, and the Preparation of Certain Symmetrical Trialkylbenzenes. J. F. Norris and D. Rubinstein. *J. Amer. chem. Soc.*, 1939, **61**, 1163–1170.—The heavy oil that separates when a solution of aluminium chloride in toluene is treated with hydrogen bromide at room temperature has a composition approaching $Al_2Br_6,6C_6H_5CH_3$. When this oil is evaporated at room temperature at 10–11 mm. pressure, the non-volatile product appears to have the formula $Al_2Br_6,C_6H_5CH_3$.

The results of the determination of the molecular weight of the oil indicate that the latter, when dissolved, was converted into toluene and the complex $Al_2Br_6,C_6H_5CH_3$.

The ethylation of benzene with ethyl chloride and ethyl bromide when the molecular ratio of Al_2Cl_6 to C_6H_5 was 1 to 1 gave yields of *s.*-triethylbenzene from 85 to 90% of the theoretical. Excellent yields of *s.*-ethyldimethylbenzene and *s.*-diethylmethylbenzene were obtained.

Evidence was obtained that the molecular ratios of Al_2Cl_6 to hydrocarbon were the determining factor in the relative proportions on which the several alkylated products were formed.

At 0° the chief product of the methylation of benzene with 3 moles of halide is 1 : 2 : 4-trimethylbenzene, whilst at 100° it is 1 : 3 : 5-trimethylbenzene.

The rearrangement of *o.*-xylene and p.-xylene to a mixture of the three isomers was studied.

The influence of temperature on the structure of the xylenes formed when toluene is methylated showed that the low temperature favours the formation of the *ortho* and *para* compounds, whilst at 100 the chief product is the *meta* isomer. In ethylation the product was largely *meta* at the two temperatures.

Detailed directions are given for the preparation of the symmetrical tri-derivatives because the methods give much higher yields and purer products than have previously been obtained. T. C. G. T.

1360. Some 1 : 4-Diaryl-1 : 3-*cyclo*pentadienes. N. L. Drake and J. R. Adams, jun. *J. Amer. chem. Soc.*, 1939, **61**, 1326–1329.—A description of the preparation of three 1 : 4-diaryl-1 : 3-*cyclo*pentadienes by the condensation of methyl aryl ketones and β-arylpropionates in the presence of sodium ethoxide and of several derivatives of these dienes. T. C. G. T.

1361. Low Temperature Dehydrogenation of Hydroaromatic Rings. R. T. Arnold and J. C. Collins. *J. Amer. chem. Soc.*, 1939, **61**, 1407–1408.—A low temperature procedure for the dehydrogenation of hydroaromatic rings by chloranil is discussed and the advantage of this method over selenium dehydrogenation is pointed out. T. C. G. T.

1362. Rearrangement of 1 : 3-Dimethyl-4-*t.*-Butylbenzene with Aluminium Chloride. L. I. Smith and H. O. Perry. *J. Amer. chem. Soc.*, 1939, **61**, 1411–1412.—1 : 3-Dimethyl-4-*t.*-butylbenzene has been prepared and characterized. This hydrocarbon, when treated with aluminium chloride, rearranges into the 1 : 3 : 5-isomer, which is the only one of the isomers obtained from *m.*-xylene, *t.*-butyl chloride, and aluminium chloride.

In view of the orienting effects of the two methyl groups in *m.*-xylene, and the rearrangement described in the paper, it is suggested that in the Friedel-Crafts synthesis of 1 : 3-dimethyl-5-*t.*-butylbenzene, the *t.*-butyl group first enters the 4-position, giving the 1 : 3 : 4-isomer, which is then rapidly rearranged by the catalyst to the 1 : 3 : 5-compound. T. C. G. T.

1363. Condensation of Alcohols, Ethers, and Esters with Aromatic Hydrocarbons in the Presence of Aluminium Chloride. J. F. Norris and B. M. Sturgis. *J. Amer. chem. Soc.*, 1939, **61**, 1413–1417.—Toluene, ethylbenzene, *t.*-butylbenzene, and *t.*-butyl-*m.*-xylene-(1, 3, 5), have been prepared by the condensation of primary aliphatic alcohols with aromatic hydrocarbons by means of aluminium chloride.

Condensation of benzene, in the presence of $AlCl_3$, with other oxygen-containing compounds is also described. T. C. G. T.

1364. Addition of Halogen and Acetoxyl to Ethylene. F. C. Weber, G. F. Hennion, and R. R. Vogt. *J. Amer. chem. Soc.*, 1939, **61**, 1457–1458.—β-Chloroethyl acetate and ethylene dichloride are the chief products when ethylene is chlorinated in acetic acid, acetic anhydride, or methyl acetate. T. C. G. T.

1365. Addition of Hydrogen Halides to *cis*- and *trans*-2-Pentene. M. S. Kharasch, C. Walling, and F. Mayo. *J. Amer. chem. Soc.*, 1939, **61**, 1559–1564.—Various workers results upon the addition of hydrogen bromide to 2-pentene seem to indicate different ratios of products depending on the condition of addition and the source and previous treatment of the pentene employed.

cis- and *trans*-2-Pentenes have been prepared and the addition of HCl and HBr has been investigated. The ratio of product has been found to be independent of the conditions of addition and the isomer of 2-pentene employed. T. C. G. T.

1366. Hydrogen Fluoride as a Condensing Agent. VI. The Alkylation of Benzene with compounds containing an " Allylic " Group. J. H. Simons and S. Archer. *J. Amer. chem. Soc.*, 1939, **61**, 1521–1522.—Hydrogen fluoride has been used to obtain reaction between a number of " allylic " compounds and benzene. Allyl benzene and 1 : 2-diphenylpropane has been prepared from allyl alcohol and 1 : 2-diphenylpropane from allyl benzene. T. C. G. T.

1367.* Studies in Water-in-Oil Emulsions. Part II. The Interfacial and Surface Activities of Magnesium and Calcium Oleates and the Rôle Played by these Soaps in the Stabilization of Water-in-Oil Emulsions. R. C. Pink. *J. chem. Soc.*, April 1939, 619–621.—The author has measured by the drop-weight method the effect of the lowering of interfacial tension between benzene and water produced by magnesium and calcium oleates. The effect is very marked, but surface tension shows a slight increase. The author observes that with soaps of multivalent metals the reduced interfacial tension permits easy subdivision of the water in the oil, with consequently less tendency to separation, and that the film of hydrated soap precipitated at the interface prevents coalescence of the globules, some of which are simply entangled in the precipitated soap ; the latter is much more readily wetted by oil than by water, which fact is apparently the type-determining factor. R. D. S.

1368.* Mechanism of Addition to Double Bonds. Part X. Kinetics of Association of *cyclo*Pentadiene. H. Kaufmann and A. Wassermann. *J. chem. Soc.*, May 1939, 870–871.—Continuing the investigations into the reaction $2C_5H_6 \rightleftharpoons C_{10}H_{12}$, the authors have measured the velocity coefficients in polar and non-polar solvents and in the gas phase. The velocity coefficients in these are all approximately the same, as are the activation energies and the kinetic A values. The latter are the products of the steric factors and the bimolecular collision numbers. Steric factors being assumed equal, the collision number in polar and non-polar solvents and in the gas phase is of the same order of magnitude. R. D. S.

1369.* Palladous Chloride as a Dehydrogenating Agent. G. W. Cooke and J. M. Gulland. *J. chem. Soc.*, May 1939, 872–873.—Attempts were made to dehydrogenate hydroaromatic and reduced heterocyclic compounds by means of palladous chloride with only fair results generally, but while the conclusion is that palladous chloride is unlikely to find general application as a dehydrogenating compound, in some cases it may perhaps prove more acceptable than selenium, especially where small quantities of material only are available. Examples are given in which palladous chloride oxidizes to carboxyl a methyl group attached to the benzene ring. R. D. S.

Analysis and Testing.

1370.* Physical and Chemical Properties of Petroleum Fractions. H. T. Rall and H. M. Smith. *Industr. Engng Chem. Anal.*, 1939, **11** (7), 387.—An apparatus is described which permits exact and rapid introduction of accurately weighed quantities of viscous-oil samples into a cryoscopic molecular-weight apparatus by diluting the sample with a known quantity of solvent from the same source as used for the molecular-weight apparatus. The molecular weights of several test oils were determined; these included a kerosine, an aromatic oil, a bright stock, and an acetone extract of a lub. oil. A comparison of the results showed that the error caused by dilution will not exceed 2%. Data obtained in the determinations of the molecular weights of three highly viscous samples are given. G. R. N.

1371.* A Microviscometer. J. R. Bowman. *Industr. Engng Chem. Anal.*, 1939, **11** (7), 409.—The method employed in this microviscometer depends on the rate of fall under gravity of a short segment of liquid contained in a longer capillary. The tube is vertical, straight, and of uniform bore—a length of 25 cm. was found to be convenient. It bears three etched marks, two near the bottom and one near the top. A glass vapour jacket is sealed around the tube. Various refinements of the apparatus are described. The instrument gives better than 4% absolute accuracy and a precision within 0·1% in the range from 2 to 10,000 centistokes. The method is regarded as simple and rapid, and requires only one drop of the sample. G. R. N.

1372.* Laboratory Fractionating Column. G. F. Reyling. *Industr. Engng Chem. Anal.*, 1939, **11** (8), 419.—Small packed laboratory fractionating columns with constricted bases often tend to become filled with condensed liquid, which cannot return to the flask because of the pressure of the rising vapours. Consequently the column fills with liquid and fails to operate properly when near its maximum capacity. This condition may be alleviated by placing a glass tube, bent in the shape of an open hook, in the bottom of the column. This arrangement permits return of the condensed liquid to the flask, and at the same time allows the vapours to pass unhindered to the top of the column. G. R. N.

1373.* Greaseless High Vacuum Valve. R. H. Crist and F. R. Brown. *Industr. Engng Chem. Anal.*, 1939, **11** (7), 396.—A more rugged and compact modification of the Ramsperger valve is described. A metal diaphragm is used with silver chloride as the seating surface. The leak through this valve as measured by a McLeod gauge was generally less than 0·0001 mm. per hour. G. R. N.

1374.* Determination of Neutral Oil and Tar Acids in Phenolic Compounds. E. Field and L. Steuerwald. *Industr. Engng Chem. Anal.*, 1939, **11** (8), 426.—In the estimation of the neutral oil the sample is refluxed with 10% caustic soda in a described procedure. The insoluble material plus a small amount of the soda extract are suitably transferred to a Babcock bottle and centrifuged to give an accurate line of separation. Results show an accuracy of 0·1% on a variety of cresylic acids and on blends containing known amounts of neutral oil. After criticizing the U.S.D.A. method for estimating tar acids, the authors describe their " liberation " method, in which the sample is extracted with 20% caustic soda in the presence of petroleum ether. The tar acids are liberated by use of 25% sulphuric acid and are washed/recovered with the aid of sulphuric acid of specific gravity 1·5–1·51. The procedure, though lengthy, gives an accuracy of 0·2% as demonstrated by the results obtained. It is held that specifications formulated on the basis of these improved methods will represent quality which is at once rigid and dependable. G. R. N.

1375.* Solubility Characteristics of Tars and Pitches Produced by Coal Hydrogenation. M. L. Fein, A. Eisner, H. M. Cooper, and C. H. Fisher. *Industr. Engng Chem. Anal.*, 1939, **11** (8), 432.—The determination of the insoluble matter in hydro tars and pitches was studied according to the various physical principles involved and to the various methods recommended. The following procedure was finally adopted. About 10 gm. of sample are weighed into a 250-ml. centrifuge bottle and heated to about 90° C. Twenty volumes of 7% cresol-tetralin (previously heated to about 90° C.) are added slowly with manual stirring. The resulting mixture is stirred mechanically for approximately 3 min. and then centrifuged at about 2400 r.p.m. for about 20 min. The supernatant layer is decanted and 150–200 ml. of benzene at room temperature are added. The solid residue is broken with a stirring rod, after which the mixture is stirred mechanically for about 3 min. and centrifuged. The layer of benzene is decanted, after which the bottle and residue are dried to constant weight at 110° C. (2–3 hr). 48 References. G. R. N.

1376.* Laboratory Ageing Tests for Lubricating Oils. F. Jostes and A. Hann. *Oel u. Kohle*, 1939, **15**, 515–518, 533–537.—The behaviour of a lubricating oil in one engine is so greatly dependent on the design of the engine and the fuel used, that it is very difficult to infer its behaviour in any other engine. Still more difficult is it to correlate laboratory ageing tests with actual performance in the engine. Laboratory methods not only show considerable differences in the fixed conditions (temperature, oxygen- or air-rate, catalysts, etc.) under which the ageing is carried out, but they also differ in that changes in different properties of the oil (asphalt or resin content, acidity, viscosity, etc.) are used as the measure of the degree of ageing. Further, even if one particular method is used, by observing the change in different properties, contradictory results for the stability may be obtained.

In the author's opinion, the difficulties arise from the fact that the oil is usually aged for a given time and the alteration in physical properties measured. He suggests that a much sounder method is to find the time taken to effect given alterations in these physical properties.

The Indiana oxidation test is based on this conception, as the reported figure is the time taken for the formation of 100 mg. asphalt/10 gm. oil. If the time is more than 50 hr., the viscosity is measured after 50, 100, 150, and 200 hr. oxidation. The author considers that the large volume of oil (300 ml.) used is cumbersome, and makes the withdrawal of truly representative samples difficult, and so suggests using 20 ml., and blowing at 650 ml./hour instead of 10 litres per hour. Comparative tests showed that the modified method gives results within the normal limits of error of the standard method, and the author used this modification of the method throughout his investigations.

In order to see if the invariants had an appreciable effect on the degree of oxidation, ten different lubricating oils were examined with the following results.

Influence of the Glass.—Oxidations were carried out in tubes of Duran, Jena glass, and Thuringrian glass; no appreciable difference was found, so all the remaining work was done with Jena-glass apparatus.

Influence of Temperature.—Oxidations were carried out at the standard temperature of 172° C., and at 200° and 240° C. The results show that, in general, the relative positions of the curves are unaltered by increase in temperature. A table is given showing the order of stability of the ten oils as indicated by the time taken to form 5, 10, and 15 mg. sludge/gm. oil at each of the three temperatures. This shows that the least reliable evidence is that given by the times of formation of 5 mg. sludge/gm. oil at the lowest temperatures. Accordingly the author suggests the further modification of the test in that the temperature of oxidation be raised to 200° C., and that the reported figure be the time taken to produce 15 mg. sludge per gm. oil.

Influence of the Air-rate.—The test was run at 172° C., with the air-rate 1000 ml./hour in place of 650 ml./hour. No important alteration in the grading of the oils was observed, the effect being the expected one of accelerated ageing.

Further oxidation experiments were carried out on (1) brown-coal paraffin wax; (2) Fischer paraffin wax; (3) octadecene; (4) oleic acid; (5) oleic alcohol; (6) synthetic oil; (7) Edeleanu extract from a light Roumanian machine oil, deasphalted with fuller's earth; (8) topped Wietz crude oil, deasphalted with propane, extracted with furfural, and twice treated with fuller's earth.

The first four gave no product insoluble in normal benzine after 100 hr. oxidation at 172° C. The neutralization value showed a large increase, especially in (1) and (3), indicating that the oxidation was mainly of hydrocarbon to carboxylic acid. (5) gave semi-solid products of which 37% was insoluble in normal benzine, but this residue was not asphaltic, consisting of other oxidation and polymerization products. (6) merely became viscous, giving no asphaltic precipitate with normal benzine. (7) had become a viscous tar after 48 hr.

It will be noticed that hydrocarbons, whether aliphatic, aromatic, or naphthenic, oxidize mainly to acids, whilst oils containing petroleum resins oxidize to asphaltic matter. Further experiments showed that the addition of resins to (6) caused the treated oil to form asphalt on oxidation, whilst untreated no asphalt is produced. It seems therefore that the ageing properties of lubricating oils are, in general, dependent on the content of petroleum resin, and not on the composition of the hydrocarbon mixture. T. T. D.

1377.* Motor Methods of Testing Fuels and Lubricants. A. v. Philippovich. *Oel u. Kohle*, 1939, **15**, 551–556.—Agreeing with the necessity of engine tests for rating gasolines, the author considers the C.F.R. motor method lacking in several respects, criticizing the bouncing-pin method of recording knock and the fact that the determinations are carried out at only one mixture strength and at only one temperature. The advantage of the octane number lies chiefly in the world-wide use of the C.F.R. engine, and to retain this the author suggests that the present method of testing be extended to give results of greater usefulness.

To determine the instant at which knocking starts, the author stresses the necessity of an automatic indicator, preferably recording the second differential of the pressure with respect to time. He further suggests that the octane number should be determined with the mixture temperature at 86° F., 122° F., and 212° F., as well as at the standard temperature of 300° F., the mixture strength being as specified in the ordinary C.F.R. motor method. From these figures, diagrams can be drawn showing the variation of octane number with temperature. The author states that four types of diagram are obtained : (1) aromatic, (2) olefinic, (3) alcohol mixtures, (4) paraffinic. He therefore suggests that in reporting the octane number of a gasoline the appropriate suffix should be used, *e.g.*, O.N. 73_2, signifies that the fuel has an octane number of 73 and that its temperature variation is of the olefinic type.

Once these diagrams have been obtained, the octane number at any required temperature can be deduced from a measurement at any other temperature, provided the composition of the fuel is known. Further, a more genuine forecast of the behaviour of a fuel in an engine is possible, as the octane number at the running temperature is readily deducible.

The deficiencies in the motor method are implicitly admitted in many specifications for aviation gasolines which require more direct information regarding the behaviour of the fuel in the engine—*e.g.*, variation of mean effective pressure with excess air at different temperatures. The author considers that these methods give results of much more significance than the mere determination of the octane number.

Turning to the testing of lubricating oils, the author again prefers actual engine tests, and suggests that a useful method is to determine the time required for ring-sticking to occur in an engine run under fixed conditions at different temperatures. In this way the stability of oils at different temperatures is obtainable, and thus the most suitable oil for use in any particular engine the working temperature of which is known, can be deduced. T. T. D.

See also Abstracts Nos. 1378 and 1379.

Gas, Diesel and Fuel Oils.

1378.* Estimation of Ignition Qualities of Diesel Fuels in the Laboratory. G. Vorberg. *Oel u. Kohle*, 1939, **15**, 497–499.—The following methods are used to obtain indications as to the ignition properties of diesel fuels : (1) hydrogen content ; (2) aniline point ; (3) diesel index ; (4) cetene number (by Marder's density method) ; (5) cetene or cetane number determined on either the I.G. or C.F.R. test-engine.

The object of this paper is to correlate the results obtained by the first four of these methods with the last, which is considered the only intrinsically reliable one. Tests were carried out on forty-two samples of widely differing origin, including oils obtained by the hydrogenation of coal, as well as of petroleum origin. The results show that all these methods give figures which correlate well with those obtained in the test-engines, except the first, and the author maintains that the hydrogen content is not a reliable guide to ignition properties. He suggests that methods (3) and (4) are very valuable for obtaining a rapid and reasonably accurate indication of these properties. T. T. D.

1379.* Simple Motor Method for Testing Diesel Fuels. H. Neumann. *Petrol. Z.*, 1939, **35**, (33), 614–621.—A new laboratory method for testing diesel fuels comprises the use of a very simple inertia indicator built into the injector needle of diesel engines for recording the ignition impulse and a receiving device connected with the crankshaft. The resulting accuracy compares well with the precision obtainable in the timing of the valve gear of the engine itself, and the ignition lag and cetene number of the fuel are easily established. E. W. S.

Lubricants and Lubrication.

1380.* Trends in Lubricating Oil Manufacture. G. A. Fitzgerald. *Petr. Engr*, Mid-year 1939, **10** (10), 105–110.—The motor industry being the biggest market for lubricating oil, changes in engine design have had a big influence on the lubricating-oil industry. Closer clearances and higher temperatures have necessitated lighter oils of greater stability and better temperature/viscosity characteristics. A demand for improved low-temperature fluidity has stimulated solvent dewaxing processes. Of these the benzol/ketone process represents two-thirds of the world total capacity. Wax removal on this method is substantially complete, and an oil with a cloud-point almost as low as the pour-point is produced. The propane process combines dewaxing and removal of high-viscosity resins to yield a lighter and more stable oil. Solvent-extraction processes have made possible the production of premium-grade oils from mixed-base and naphthenic crudes, as well as from paraffinic crudes. Of the five leading processes, furfural, Duo-Sol, phenol, SO_2-benzol and chlorex, furfural is the most popular, probably owing to the normal extraction temperatures, 150–250° F., used. A feature of all the solvent methods is the application of instrument control, which makes for uniformity of production and more economic operation. High vacuum distillation is also used for the removal of resins and asphalt—with some oils it is more economical than solvent extraction.

In the decolorization of motor oils increased use of activated earth and bauxite has contributed to a fall in the consumption of fuller's earth. The accomplishment of decolorization in combination with distillation, with consequent saving of heat, is claimed. The demand for extreme-pressure lubricants has resulted in the production of oils containing sulphur, chlorine, and other active agents. Improvement in diesel-engine lubricants is evidenced by a steady increase of diesel-engine installations. Laboratory development of solvent extraction and molecular distillation has contributed to a better knowledge of the chemical composition of lubricating oil.

The catalytic effect of metals on the deterioration of oils has been closely studied and effective inhibitors have been produced. Other dopes have proved effective in depressing pour-point, preventing varnish formation and bearing corrosion, increasing oiliness, and raising viscosity index. R. J. E.

1381.* Lubricating Oils for Automobile and Tractor Engines. P. A. Khokhryakov. *Neft. Khoz.*, June 1939, **20** (6), 37–39.—Properties of engine oils at present produced in the U.S.S.R. by sulphuric acid treatment are considerably below those required. Production of high-grade engine oils depends on output of selective-solvent-treated oils and development of a suitable method of lowering their pour point. These engine oils should agree with S.A.E. specifications, especially regarding viscosity.

Methods of reducing pour points of sulphuric acid-treated oils should be developed at an early date so as to ensure the supply for the winter season 1939–1940. L. R.

1382.* Oxidation Tests with Roumanian Turbine Lubricating Oil. C. Candea and C. Manughevici. *Petrol. Z.*, 1939, **35** (33), 611–614.—It is possible to show by means of a laboratory test that different dopes reduce the tendency of the oil to combine with oxygen. The following materials were added as dopes: thymol, anthrazene, naphthalene, toluidine, and *para*-methyl- *para*-diamido-diphenylmethane. The ageing of turbine oil in operation being due mainly to oxidation, the authors consider it advisable to use such chemicals as stabilizers. E. W. S.

1383.* Melting Point of Solid Greases. D. S. Velekovskii, I. P. Lukashevich, and F. L. Borshchevskaya. *Refiner*, 1939, **18** (8), 312.—Structural changes brought about by details of production have a pronounced effect on the Ubbelohde melting point, but have no effect on the performance of the greases at high temperatures. Sodium base greases are heterogeneous systems, the thermal behaviour of which is greatly affected by details of production. In calcium base greases the effect of the structural peculiarities is balanced by the water present. There is no simple relation between the structure of a grease and its stability toward thermal action. The melting point is determined, other factors remaining constant, by the content of soap, rather than by the viscosity of the mineral oil used. The resins exert a stabilizing and peptizing effect. A rise in the valence of the soap base lowers the dropping temperature, the duration of melting, and the thermal stability of the grease. Increasing saturation of the hydrolysed oil or fat raises the thermal stability, the dropping temperature, and the duration of melting. G. R. N.

1384.* " Seizure Delay " Method for Determining Seizure Protection of E.P. Lubricants. H. Blok. *J. Soc. aut. Engrs*, 1939, **44**, 193–200.—Considerations of the influence of temperature on the testing and performance of so-called E.P. lubricants have so far been neglected according to this author. It is considered that in operation a " temperature flash " is superimposed on the average " bulk " temperature of gears. This " temperature flash " may last for only 0·0001 sec. and is thought to be the governing criterion for the failure of the lubricant. " Bulk temperature " should be controlled when testing such " extreme-temperature-pressure " lubricants; the failure of conventional testing methods is attributed to lack of this control. Testing technique consists in determining " seizure delay " using the familiar " Four Ball Top." This is measured by the load to cause seizure 2·5 sec. after starting the test. After seizure, equilibrium is again reached under high loads, but under still higher loads welding may occur. The amount of wear is a measure of the intensity of seizure. One peculiar phenomenon was observed in this, as in the S.A.E. machine—namely, that " incipient seizure " can occur with some E.P. lubricants at loads not much higher than those which cause failure with straight mineral oils. This is considered to be due to the fact that the temperature rise under such conditions is not sufficient to render the additive agent active. No correlation was found between results on the S.A.E. and Timken machines operated conventionally and on the " Four Ball Top " operated by the " seizure-delay " method. This is considered to be due to the uncontrolled variable of bulk temperature in conventional methods of test. Better correlation was obtained when the S.A.E. machine was operated by the seizure-delay method. Reasonably good correlation was established between results by the " Four Ball Top " using the seizure-delay method, and the lowest load at seizure in a four-square set-up of Chevrolet hypoid rear axles operated at a speed equivalent to 40 m.p.h. on the road. The temperature of the oil in the rear axle was controlled to 95–100° C. It is claimed that with the " Four Ball Top," even " strong "-type hypoid lubricants can be evaluated. The appendix to the paper is devoted to theoretical considerations. Protection against seizure is shown to be dependent on temperature at seizure and friction before seizure. The reason for taking 2·5 sec. as the time in the seizure-delay method is that this time is too short for bulk temperature to differ appreciably from ambient temperature, and the difference can therefore be ignored, and only temperature flash be considered. Practically this temperature flash can be measured by making the balls of dissimilar metals. J. L. T.

1385.* Symposium on Varnish in Engines. F. F. Kishline. *J. Soc. aut. Engrs*, 1939, **45** (2), 321–324.—Incipient varnish causes partial or intermittent sticking of valves and piston rings; in acute form varnish has been known to stick engines so that they could not be turned over with the starter. The replies of many S.A.E. members to a questionnaire on this subject reveal a wide divergence of views. Sticking-up of the engine is described as occurring when the engine is permitted to idle after normal driving, and usually happens in relatively new engines; in at least some such cases the engines were not operated under any abnormal conditions.

Several contributors were of the opinion that the varnish problem had come from a change in the character of oils. In four replies it was stated that varnish could be rapidly produced at sump temperatures above 280° F., whilst two others were equally emphatic that high temperatures were not the primary cause. One attributed the trouble to reduced clearances in 1939 engines, whilst two others ruled out small clearances as a likely cause. One reply suggested that infrequent oil changing might be a contributory factor.

Summarizing, the author concludes that varnish is not partial to any particular engine design; it would appear that time and temperature are principal items which govern the condition, and the temperature may or may not be the sump temperature. Possibly the rate of oil circulation over hot surfaces is a factor. Closer co-ordination between automobiles and lubricants is advocated. K. T. A.

Asphalt and Bitumen.

1386. Type of Aggregate Gradation for most Suitable Asphaltic Mixtures. R. Vokac. *Assoc. Asph. Paving Techn. Proc.*, 1939, 86–123.—In a previous paper the authors described a method of selecting mixture composition by means of an isometric design chart, in which certain characteristics, such as compression strength and voids, may be determined for any mixture composition within the normal range. An investigation of the correlation of tests with service behaviour indicated that mixtures having compressive strengths of 575–1100 lb./sq. in. and voids of 2–6% were usually satisfactory, mixtures of lower strength were soft and tended to displace under traffic, whilst mixtures of higher strength were liable to crack owing to excessive hardness. It was also observed that the design charts could be separated into three main types, of which (1) represented mixtures in which smaller amounts of bitumen than usual were required and bitumen and filler contents varied widely with little effect on physical characteristics; (2) materials which gave satisfactory mixtures with a restricted range of bitumen and filler contents, and (3) materials which do not give satisfactory mixtures within the usual range. The design-chart type was found to be dependent on the aggregate gradation, and examination of a large number of curves indicated that type (1) aggregates show a wide range of particle sizes evenly distributed and a larger percentage of coarse aggregate than the other types, whilst type (2) is intermediate. It is shown that the type can be identified by the middle 50% of the gradation curve and, when a series of sieves is selected in which the mesh opening of one sieve is twice that of the next smaller sieve, if the percentage passing the various sieves is plotted against the log of the opening the portions of the curve corresponding to the middle 50% approximates to a straight line. The average percentage passing between two adjacent sieves in the middle 50% may therefore be determined from the slope of the curve. The gradation type can be decided as follows. If no single fraction contains more than 23% of aggregate and any two adjacent fractions not more than 46%, the aggregate is type 1; if any single fraction contains more than 23% and any two adjacent fractions contain 60–75%, the aggregate is type 2; if any single fraction contains 48% or more of the aggregate and/or if any two adjacent fractions contain more than 80%, the aggregate is type 3. A. O.

1387. New Vibrating Machine for Determining Compactibility of Aggregates. J. T. Pauls and J. F. Goode. *Assoc. Asph. Paving Techn. Proc.*, 1939, 124–145.—The paper describes an apparatus for determining the maximum attainable density of aggregates. This consists briefly of a vibrating table on which is mounted a cylinder having one plunger fitting into the bottom and another fitting into the top, the aggregate being contained between the two plungers. The cylinder is vibrated for 20 min. and the initial and final heights are measured by means of a micrometer dial gauge. When testing aggregates having less than 35% passing a No. 10 sieve, 50–70 ml. of kerosine are added to 750 gm. of aggregate to prevent segregation; aggregates with more than 35% but less than 50% should be tested with and without kerosine, and the highest value reported; aggregates with more than 50% should be tested dry. Tests made on various types of aggregate showed that densities obtained in circular track sections constructed with low-cost materials agreed closely with those obtained by the vibrating test; other methods of compaction generally gave lower densities. Hot plant-mix materials cut from road surfaces had lower densities than those obtained by vibrating aggregates without binder. Results are given for various types of aggregates. A. O.

1388. Comparison of Methods of Specifying Aggregate Gradations. S. Walker. *Assoc. Asph. Paving Techn. Proc.*, 1939, 146–159.—The author considers the relative merits of the method of specifying aggregate gradation in terms of cumulative percentages as opposed to separated percentages. It is thought that the methods are widely different in significance and are not readily comparable. The cumulative method is preferred, since the specification can be expressed in graphical form and tolerances are more easily decided. A. O.

1389. Composition and Testing of Asphalts of the Slow Curing Type. J. Zapata. *Assoc. Asph. Paving Techn. Proc.*, 1939, 160–194.—The paper deals with an investigation of the significance of tests applied to slow-curing liquid asphalts. Five asphalts were separated by precipitation with propane and hexane into asphaltenes, resins, and oily constituents from which a series of 131 asphalts of known composition was prepared. These asphalts were then tested according to the methods of the A.S.T.M. and the American Association of State Highway Officials, stripping and laboratory weathering tests also being made. Results obtained indicated that a definite relationship exists between composition and specific gravity, asphaltenes having a greater effect on specific gravity than resins. Resistance to stripping was found to improve with increase in specific gravity, and samples not containing asphaltenes exhibited low resistance to stripping. Absolute viscosity increased with increasing asphaltene or resin content, and the author is of the opinion that viscosity ranges in current use are not sufficiently high to enable high resistance to stripping to be obtained. Loss on heating increased with increase in oily constituents, and it is thought that the loss was due entirely to oily constituents. Evaporation is also believed to be the principal cause of increase in consistency in the 100 penetration residue test, and this test is believed to be of value in determining rate of change in service. In ductility tests at 4° C. unsuitable proportions of asphaltenes and resins or of resins and oily constituents resulted in low ductility. The fluidity factor appeared to differentiate between asphaltic and paraffinic types, and permitted of the identification of cracked residues. Positive results were obtained in the Oliensis spot test on all blends of cracked oils and on certain blends of non-cracked fractions; the original oils from which the latter were prepared gave negative results. The test for insolubility in petroleum naphtha is believed to be a useful supplement to specific gravity as an indication of resistance to stripping. It was found that results obtained with 88° Beaumé naphtha were not always consistent and did not agree with the results when using hexane, the solvent normally employed by the author. In the weathering test no definite correlation was observed between rate of increase of asphaltenes and composition, and this type of test was considered unsatisfactory. In an attempt to correlate the various tests with service behaviour a road surface prepared with S.C.-7 road oil was kept under observation for 60 weeks; samples of the mixture were taken immediately after laying and every week for two months and at the end of sixty weeks. The bitumen was recovered by Abson's method and examined for asphaltene content, resins and oily constituents, and refractive index and viscosity at 140° F. The results obtained showed that the residue at the end of 60 weeks was not appreciably hardened and corresponded to 150/200 penetration; it is therefore thought that it would be many years before the penetration reaches 100. It is also considered that evaporation is the principal cause of hardening. A. O.

1390. Asphalt Paving Equipment. F. S. Gilmore. *Assoc. Asph. Paving Techn. Proc.*, 1939, 195–216.—A review is given of developments in the use of plant and equipment in the maintenance of asphalt paving. A. O.

1391. Surfacing Asphalt Pavements for High Light Visibility. C. A. B. Halvorson. *Assoc. Asph. Paving Techn. Proc.*, 1939, 217–241.—It is pointed out that under city lighting objects are seen by light reflected from their vertical surfaces, and details and colour contribute to visibility, whereas on arterial roads, owing to low illumination, objects are generally seen in outline contrasted against the brighter background. Road surfacings absorb light in varying degrees, and certain surfacings give diffuse reflection, others specular, whilst some lack uniformity of reflection over large areas necessary for proper visibility at night. Pavements and paving materials generally change in light reflection coefficients and characteristics when wet, and brightness uniformity varies greatly with the specific intensity, direction, and angle of the applied light. The brightness appearance of some pavements is also greatly affected by differences in surface texture and finish, under the variation in quality and direction of daylight. In tests on a model road having a wet, smooth, black surface, two light-coloured objects with matte surfaces of identical reflectivity were placed on the pavement; one of these appeared black against the longitudinal image of the source of light reflected on the pavement, whereas the other appeared white, being illuminated by light directed away from the camera. The surrounding pavement appeared dark owing to the direction of light falling on the wet surface, and a dark object would be practically invisible in this area. Under the same conditions but with a dry diffusing or matte surface the objects still appear black and white, but since the pavement reflections appear as transverse areas they are clearly visible. Pavements of fine texture, irrespective of their dry diffuse reflectance factor, when wet reflect sources of light as in the first experiment. This is due to the small particles being submerged by a film of water. Open-surfaced pavements usually conduct the water away and prevent formation of a film, and when the aggregate has reasonably high diffuse reflectance of the wet emergent surface behaves as in the second experiment. In experiments made to determine how visibility and economy are affected by difference in behaviour of two types of pavement differing widely in diffuse reflectance under artificial lighting, an open-type macadam pavement with a dark surface of low diffuse reflectance value was illuminated by shading-type luminaires 120 ft. apart, staggered and fitted with 400-watt Mazda lamps. Under this lighting the pavement appeared as small spots of brightness alternating with large areas of darkness. Four targets having surfaces painted vertically half white and half black were placed in the traffic lanes at distances of 100 and 200 ft. from observers. The white portions were easily seen in contrast with dark areas of pavement, but although the black portions were approximately the size of a human being, it was only possible to see them in two cases; these were silhouetted against the bright areas of pavement. The road was then surface dressed with ½–¾ in. granite chippings, producing a matte surface, and although other conditions remained as before, the pavement now appeared reasonably bright, and black portions of the targets were clearly visible. This change resulted in improvement in brightness of about threefold. Adjustments to light distribution of the luminaires gave a relative improvement in the shadow of about 4 to 1. The information obtained indicated that properly designed lighting giving uniform illumination on a matte surface from overhead would produce uniform brightness from any angle. In bright sunlight the pavement was appreciably less bright when facing the sun than the original pavement. The pavement was also uniformly bright when wet and visibility distance with automobile headlamps was increased several times. Visibility of objects was high and the pavement appeared uniformly bright under a cloudy sky.

A. O.

Special Products.

1392. Propane Precipitation of Petroleum Resins. P. T. Graff. Symposium on Plastics and Resins from Hydrocarbons. Amer. chem. Soc. Meeting, Boston. September 1939, pp. 5–14.—The residue from the distillation of crude petroleum may be separated into asphalt, resins, and lubricating-oil fractions by precipitation with liquid propane. This generally involves a two-stage process, the first stage being concerned with the separation of asphalt, whilst the second effects the separation of the resins.

These petroleum resins contain those hydrocarbons or colour bodies which are adsorbed on clay or earths in the refining of lubricating oils. They are similar to asphaltenes, except that they are not oxidized or combined with sulphur, and are thus soluble in petroleum ether.

This precipitation of resins is used commercially to reduce the duty of clays in decolorizing lubricating-oil stocks. The products obtained may vary greatly in composition, depending on the nature of the residual oil and its previous history. They may be very narrow cuts on the crude, and consist essentially of bodies with a high C : H ratio. On the other hand, with broader cuts they may be associated with high-molecular-weight lubricating fractions.

Diagrams illustrating the effect of propane de-resining are included in this paper.
D. L. S.

1393. Production of Petroleum Resins. S. C. Fulton and A. H. Gleason. Symposium on Plastics and Resins from Hydrocarbons. Amer. chem. Soc. Meeting, Boston. September 1939, pp. 15–28.—The paper first deals with the resins which occur naturally in petroleum, and those formed during cracking processes.

Two methods are given for recovering the latter from cracking coil tar : (1) the tar is vacuum-distilled to a residue with softening point 105–115° C. This residue is extracted with a refined petroleum distillate, the solution filtered from finely divided and suspended asphaltenes, acid treated, and finally distilled to recover the resins. (2) Tar is distilled to a low-melting-point pitch, which is treated with anhydrous aluminium chloride under condensing and polymerizing conditions without dilution. Treatment with sulphuric acid and subsequent neutralization follow, after which the resins are separated by distillation under reduced pressure.

These two classes of resin are not of good quality as regards colour and melting point, and a better-quality product can be synthesized from light distillate oils by the Formolite process. The most satisfactory resins as regards colour and melting point have been prepared by refluxing a mixture of aromatic hydrocarbons, formaldehyde, and zinc chloride in glacial acetic acid. The four components of this reaction are discussed separately, and the influence of each on the final product is described.

Resins prepared from highly aromatic light petroleum distillate and formaldehyde are amber to pale yellow in colour, are very brittle, and have melting points up to 140° C. They can be used with success in paints and varnishes.

As regards heavy distillate oils, in addition to formaldehyde condensations, two other methods of resinification have been satisfactorily employed. These involve chlorination and either thermal dechlorination or condensation with metallic halides.

(*a*) *Resins Produced by Chlorination and Dechlorination.*—Cracked petroleum tar is distilled under vacuum conditions and the distillate chlorinated slowly at room temperature until 10–15% of Cl_2 has been absorbed. The chlorinated distillate is then heated and distilled, and the bottoms, after some cooling, are agitated with a selective solvent. This is then distilled off to recover the resin.

(*b*) *Chlorination and Condensation with Metallic Halides.*—As in (*a*), the distillate is chlorinated. It is then treated with 2–10% of a metallic halide such as aluminium, ferric, or zinc chlorides at 75–100° C. for about 10 hr. After decomposition of the catalyst, unreacted material is removed by distillation to 320° C. at 1 mm. pressure. The residue is selectively extracted to recover the resins.

From all these processes resins can be obtained with physical and chemical properties such as to make them appear interesting for a variety of purposes. D. L. S.

1394. The Properties of the Polybutenes and their Uses in Petroleum Products. R. M. Thomas, J. C. Zimmer, L. B. Turner, R. Rosen, and P. K. Frolich. Symposium on Plastics and Resins from Hydrocarbons. Amer. chem. Soc. Meeting, Boston. September 1939, pp. 29–51.—The production of polybutenes of molecular weights ranging from 25,000 to 400,000 and higher from *iso*butene is now an established process. These polymers may be classified according to their molecular weights as determined by the Staudinger method, which depends on the increase in viscosity of a solvent due to the solution of the polymer.

The polybutenes vary from liquids to white, tough, elastic solids having a H : C ratio 2·0. They are odourless and tasteless when pure, and have a specific gravity of approximately 0·9, which varies slightly with the molecular weight. They are more resistant to heat, ultra-violet light, and mechanical working than rubber. They are soluble in liquid aliphatic hydrocarbons, and except for the high-molecular-weight members, in aromatics as well, and have the property of increasing the viscosity index of petroleum oils.

They are used in motor oils for improving the viscosity index, and because of their stringy properties are of great value in greases and special lubricants. A method of measuring the stringiness of blends is described. In asphalts these polymers tend to raise the softening points and increase the penetration, whilst improving the ageing properties of these products. Paraffin wax is also improved in some directions by admixture with polybutenes. Thus the low-temperature flexibility of wax can be considerably modified by this means and the adhesion of waxed surfaces rendered more satisfactory.

Amongst other uses may be included their utilization in (*a*) motor fuels, (*b*) shock-absorbing liquids, (*c*) cosmetics and medicinal preparations, and (*d*) compositions for impregnating leather. D. L. S.

1395.* Comparative Data on the Characteristics of Activated Lampblack from Methane " Carbomet No. 3." G. Vulpescu, *Monit. Pétrole roum.*, 1939, **40**, 1009–1012.—" Carbomet No. 3," a product of the Société Nationale de Gaz Méthane, is an activated carbon black the physico-chemical characteristics of which show it to be as good as the best-known American grades, tests of which are given in comparison with Carbomet No. 3. An account of the methods of testing is given in this paper. They include: (1) the heat of wetting by benzene which is a function of particle size, (2) adsorptive capacity, (3) maximum adsorption. Adsorptive capacity is measured best with diphenylguanidine (D.P.G.), which has a straight-line adsorption isotherm. The sample is treated with $0{\cdot}01N$ solution D.P.G., shaken, and then centrifuged; after titration of excess D.P.G. with $0{\cdot}01N$ hydrochloric acid the index of adsorption is calculated. Maximum adsorption is measured by passing air saturated with an organic solvent such as carbon tetrachloride through the carbon black until the latter ceases to gain in weight. R. D. S.

Detonation and Engines.

1396.* Prospects for Use of " Safety Fuels " in Spark-Ignition Aircraft Engines. F. C. Mock. *J. Soc. aut. Engrs*, 1939, **45**, 291–294.—Recent developments in fuel-refining processes have revived general interest in the subject of safety fuels, and this article summarizes the probable programme necessary before such fuels can be employed successfully in everyday service operations. As such fuels so far seem inherently to have a lower calorific value than gasoline, they start off with a handicap of from 3 to 7% in fuel consumption as compared with gasoline, and any successful development must achieve as complete combustion of both air and fuel charge in the cylinder and as accurate metering of the fuel/air ratio as are now obtained with the newer carburettor systems.

The author confines his analysis of the problem to the use of fuel injection, as with this system the two outstanding difficulties encountered—viz., obtaining adequate heat for fuel vaporization and subtracting heat from the hottest parts of the combustion chamber—are brought into mutual solution. Injection equipment at present available should be satisfactory, but to co-ordinate pump delivery with air throttling it is necessary that a scale should be provided indicating the fuel charge delivered at each position of the pump control. Satisfactory operation cannot be obtained in any present engine without changes to affect turbulence and temperature distribution. Single-cylinder engine tests should be made at all loads and speeds encountered in service, and special attention should be given to cool engine operation. Tests on a radial air-cooled engine and a Prestone cooled single-cylinder engine injecting into the cylinders and using fuel of 87 octane number have shown satisfactory results, and in the case of the latter engine it was found necessary to raise the piston crown temperature to form a " hot spot," as the combustion chamber walls ran below the boiling point of the fuel. Suggestions are made with regard to the starting problem with safety fuels, and in connection with the installation of the injection equipment. In the author's opinion the completion of the work necessary for a complete solution of the problem must be undertaken by the engine manufacturers, and would require at least 2½ years. C. H. S.

1397.* Trend in Poppet Valves. A. T. Colwell. *J. Soc. aut. Engrs*, 1939, **45**, 295–304.—Although during the past year no entirely new development has been forthcoming, many improvements upon existing performance have been achieved. These improvements have been the outcome of studies connected with grain flow, structure, forging temperature, coolants, and interior construction. In addition, 300 analyses of valve steels were examined and intensive work on twenty steels showed four of definite merit. The results of investigations dealing with sodium cooling and head designs by means of glass valves are illustrated and discussed, and chemical and physical analyses of four outstanding valve steels are given, the steels concerned being Silcrome No. 1; Silcrome XB; Silcrome X10; and Silcrome XCR. The author also discusses rotating valves, valve guides, valve-seat inserts, "superfinish," and various surface treatment processes. C. H. S.

1398.* Aviation Fuels and Engines. F. R. Banks. *J. Soc. aut. Engrs,* 1939, **45,** 389–406.—The first part of this paper reviews the present position regarding aviation fuel in Great Britain, and particulars of the leading British engines are given, together with their performance on the fuels used. It is claimed that British engines can take greater advantage of the temperature-sensitive fuels, such as gasoline/benzol and gasoline/benzol/alcohol blends, than some of the typical American engines, instances being quoted where the latter engines with hemispherical two-valve heads gave trouble with overheating on such fuels, whereas a well-known British air-cooled engine with four valves per head gave no trouble. A theory is put forward to account for the differences experienced in this respect. It is believed that the greater rotational swirl given to the fuel/air charge in the two-valve head renders this type of engine more sensitive to spark-plug position, spark timing, and the fuel used than is the case with the four-valve head, in which this rotational swirl does not occur. Comparisons between temperature conditions; m.e.p.'s, and speeds of typical American and British engines are given which indicate that temperatures alone cannot account for the differences noted in respect to behaviour on these so-called temperature-sensitive fuels.

Considerable data are given on special fuels for racing purposes with which the author has had a wide experience; and particulars are given of their performance in engines used for record-breaking purposes.

Differences in British and American valve technique are dealt with, and some account is given of the new development in exhaust-valve manufacture which employs "Brightray" (an 80/20% nickel chromium alloy) for valve faces and complete valve forgings.

The advantages claimed for the single-sleeve valve by its protagonists are discussed individually, and whilst the author admits that the Bristol Co. has done an excellent job in bringing the sleeve valve to its present state of development, he nevertheless contends that this type of engine has not shown itself to be a development which demands immediate adoption regardless of all other considerations.

An important development in England in the last year or two has been concerned with the use of sintered aluminium-oxide spark-plug insulators, which were first produced by Siemens in Germany. The use of this material with fine earth wires has proved extremely satisfactory, and very long life is now being obtained with these plugs. This subject is discussed at length, and many illustrations of this type of plug and the latest type of shielded cable elbow employing bakelite insulation and 1000 ohm resistor between cable and plug are given.

The use of fuel injection is discussed, and the author thinks that this method which is now largely used in Germany should be seriously tried out, particularly in the case of large multi-cylinder supercharged engines, in which distribution difficulties are likely to be encountered.

The future development of engines must depend on the future requirements of aviation, and these appear likely to be governed by military needs for some time to come. The latter section of the paper deals with probable development for such purposes.

It would appear that engines of 2000 b.h.p. up to, say, 5000 b.h.p. are likely to be required in the next few years, and the author thinks that the liquid-cooled engine will probably be employed for these higher powers, as it offers more scope in dealing with the increased heat flow which will be encountered. Compactness of design and greater rigidity are also possible with this type of engine. There will probably be three main types—viz., the "X" engine with four banks of six cylinders each; the "H" engine, arranged either vertically or on its side; and the multibank radial, with in-line cylinder arrangement. Probable general design features of such engines are discussed, especially with regard to number and optimum size of cylinders, and the main points for and against the Diesel or C.1 engine are dealt with. Appendices deal with tests on 87 and 100 octane fuels in a Napier-Halford "Dagger" twin-cylinder test unit, and a report by the Chemical Standardization Committee of the Institute of Petroleum on Secondary Reference Fuels. C. H. S.

1399.* Precision of Knock Rating, 1936–1938. D. B. Brooks and Robetta B. Cleaton. *J. Soc. aut. Engrs,* 1939, **45,** 449–456.—The first analysis of detonation rating data on Co-operative Exchange Samples was completed by the U.S. Bureau of Standards in the report presented to the C.F.R. Detonation Subcommittee on 4th May, 1936. The Bureau was later requested to make a second analysis of the Co-operative Exchange fuels since June 1936, and the present report analyses the results obtained in 6386 tests on 136 fuels, the tests being made by over 100 laboratories.

This report shows that the accuracy of knock-rating tests when corrected to 70 octane number, and expressed as standard deviation, has improved from 0·63 octane unit in the previous analysis to 0·52 octane unit in the present analysis.

The general improvement in precision averaged over 20% on all types of fuels, but although the improvement has been large for fuels of 70 octane number, it has been nil for those over 80 octane number. Results on cracked fuels of 70 octane number showed less precision than those on straight-run fuels of the same octane number. The 1939 Research Method was definitely more precise than the older Research Method.

The effects of atmospheric and engine variables reported in 1936 are substantiated in general, and it is concluded that more attention should be given to the correction of compression ratio for barometric pressure. It also appears that better results would be obtained if not more than 100 hr. were allowed to elapse between overhauls of rating cracked fuels. Humidity control is also recommended. C. H. S.

1400.* Automobile Cooling and its Associated Problems. C. S. Steadman. *J. Instn aut. Engrs,* 1939, VII (10), 27–53.—This paper is intended to be a general survey of automobile cooling-system practice, together with associated problems. The performance of a cooling system can be conveniently expressed by the "Critical Air Temperature"; this is the maximum atmospheric temperature at which the car can be operated continuously under specified load conditions without boiling. Heat dissipation from the radiator is proportional to the difference between mean air and water temperatures across the radiator system; hence the critical air temperature (C.A.T.) can be estimated from track or chassis dynamometer tests at any convenient lower temperature. The C.A.T. of the typical large American car is 45–47° C. under full throttle conditions and approximately 20° C. under idling conditions. The latter figure proves to be fairly adequate for practical traffic conditions. For the British market a full-load C.A.T. of 35° C. is considered satisfactory.

Methods of radiator construction are reviewed, and data given regarding heat dissipation from different matrices. For maximum economic cooling effectiveness the water-pump output should ensure a temperature drop across the radiator at maximum speed full throttle of 6–8° C. This means a flow of 25–30 gal. per min. for a 30-h.p. car. The fan for a car of this size may absorb 8–10 h.p.

For optimum operating conditions the coolant temperature should be kept in the neighbourhood of 80° C. This temperature should be reached with a minimum delay after starting from cold. Various methods of thermostatic control are discussed, and it is concluded that requirements are best met by a thermostatic valve in the top water-pipe with a large by-pass to the water pump inlet. The thermostat is arranged to start cutting off the by-pass as the main valve begins to open; when the latter is fully open the by-pass is entirely closed. Under-bonnet temperatures are much lower with water-circulation control than when radiator shutters are used.

With high-powered engines an oil cooler is often necessary. Cooling of the oil may be effected by air, as with the main water system, or by means of a honeycomb matrix through which the main cooling water passes. The latter method also helps to warm up the oil when starting from cold. Oil coolers should be capable of withstanding an internal test pressure of 200 lb. per sq. in.

The author points out that the causes of overheating troubles often lie in the engine itself, such as stagnant water pockets which result in steam formation. Pressure cooling is considered to have distinct possibilities; the system is sealed by a blow-off valve and is designed to operate above atmospheric pressure at full capacity. The boiling point is thus raised, and a smaller radiator can be used. In one such system the pressure was 12 lb. per sq. in., the boiling point 117·5° C., and the radiator weight was reduced by 35%. K. T. A.

1401. The Work of the D.V.L. F. Seewald. *J. R. aero Soc.*, 1939, **43** (343), 508–545.—The D.V.L. is the central scientific aeronautical research institute in Germany. An outline is given of its constitution and its recent activities under three headings: (1) the aeroplane, (2) the engine, and (3) equipment. In regard to engine development the requirements being studied are increase in cruising altitude, performance, and range.

Since 1936 many tests have been made in the altitude chamber on liquid- and air-cooled engines, with particular reference to supercharging. It is now possible to maintain constant pressure up to 10 km. with a single stage, and without cooling of the compressed air before admission to the motor. An outline is given of the progress in development of exhaust-driven turbines with air-cooled rotors. The possibilities of sleeve-valve motors have been studied, with promising results. Investigation of long-range power units has indicated that the possibilities of the two-stroke diesel motor appear to be favourable for flight at medium altitudes; for high altitudes the ignition motor developed to give economical operation, as well as the four-stroke diesel motor, both with exhaust gas turbo superchargers, promise successful application.

Experiments on the atomization of fuel jets are illustrated by spark-discharge photographs. The velocity of the fuel drops in the centre of the jet is greater than that at the edges. It can be shown that self-ignition of the fuel takes place at the edge of the stream. K. T. A.

Economics and Statistics.

1402.* 1938 Drilling Results of Azneftedobycha and 1939 Perspectives. M. S. Shishkanov and N. Ya. Udyanski. *Azerb. neft. Khoz.*, January 1939, **19** (1), 12–15.—During eleven months of 1938 only 75% of the plan in terms of depth drilled have been fulfilled, and in place of 913 wells planned only 611 (67%) have been sunk. The number of survey wells drilled has been 55% of that planned. Percentages of plan fulfilment are given for individual trusts belonging to Azneftedobycha. Reasons for failing to reach plan figures are analyzed, the main cause being the great number of breakdowns. During the eleven months a total of 1399 breakdowns have been reported, taking 9·4% of working time.

Organization of drilling is examined in detail, and found unsatisfactory. 6·1% of total working time were lost through unexpected troubles during drilling, whilst time loss due to bad organization amounted to 13·5%. In total, 31·8% of the working time was lost. Unsatisfactory results are traced to incorrect drilling practice, resulting in a great number of crooked holes. Drilling equipment is often not appropriate—for example, bits not suited to soil hardness. Drilling mud is carelessly prepared and insufficiently screened. Modern methods, although approved, find slow introduction.

Measures for avoiding past mistakes in 1939 are indicated, including, (*a*) improved organization, (*b*) preparation of standard plans for erecting derricks, (*c*) necessity of dragging derricks wherever possible, (*d*) selection of right type of pipes, (*e*) use of three-cutting-edge bits where possible, (*f*) use of coarser-grain material for cementation, etc. L. R.

1403.* World Legislation Affecting Petroleum. Anon. *Bol. Inform. Petroleras*, June 1939, XVI (178), 60–70.—This is a continuation of a reference index to various laws affecting the petroleum industry, contained in No. 174 and subsequent numbers. H. I. L.

1404.* Project for Increasing the Customs Duties on Refined Combustible Liquids in Uruguay. C. R. V. Garzon. *Bol. Inform. Petroleras*, June 1939, XVI (178), 71–76.—Full particulars are given of the increases which have been sanctioned with a view to protecting the home refining industry. Advice is given of a contract made with the Associated Ethyl Co., Ltd., London for the supply of Ethyl Petrol of seventy-four octanes. H. I. L.

1405.* New Laws Decreed Affecting the Import, Export, Transport, Distribution and Refining of Petroleum and its Derivatives in Brazil. Anon. *Bol. Inform. Petroleras*, June 1939, XVI (178), 77–83.—This is a verbatim copy of the decree. H. I. L.

1406.* Production in the Argentine for the first six months, 1939. Anon. *Bol. Inform. Petroleras*, June 1939, XVI (178), 1–2.—The figures show a net increase over the corresponding period in 1938, of 82·15% for the Y.P.F. and of 17·85% for private companies. An interesting graph is included, showing the prices of petrol from the year 1920 ($0·35) up to the present time ($0·18). H. I. L.

BOOK REVIEWS.

Oil-Field Exploitation. By Lester Charles Uren (Professor of Petroleum Engineering, University of California). Pp. 756. 9 × 6, 386 illustrations. McGraw Hill Publishing Co., Ltd., Aldwych House, London, W.C.2. Price 36*s*. net.

The publication of this volume completes the revision and rewriting of the author's original work on Petroleum Production Engineering, published in 1925.

The important advance made in oil-field technology, thereby placing it on a much higher technical plane, has necessitated the rewriting of a large part of the original work, and it has been found necessary to divide it into two volumes, which the author (the well-known professor of Petroleum Engineering at the University of California), has subtitled as follows: (1) Oil-field Development, (2) Oil-field Exploitation. The former was published in 1935, and covers the period from the securing of the land for development purposes, up to the completion of the wells in preparedness for production. The latter volume, published this summer, covers the period up to the time the oil is ready for delivery to the market.

The author has used as his background the lectures given by him to senior petroleum engineering students, on which he has enlarged, and has drawn on his vast knowledge and experience and from the published literature on petroleum technology in the technical press, the journals of various learned societies, and from publications of the U.S. Bureau of Mines.

The industry, therefore, is furnished with a work which combines the very latest thought and practice, and which provides it with a proper perspective, together with a knowledge of its terminology, equipment, and methods.

Each volume comprises twelve chapters, is profusely illustrated, and is complete in itself.

In the present volume the author deals, at the outset, with the various principles appertaining to oil drainage, and analyses reservoir conditions and factors affecting the removal of oil from same, the discussion of the retentive forces restricting petroleum drainage, capillarity, adhesion, and flow friction, and characteristics of oil and gas wells. The following five chapters deal entirely with the recovery of oil and the various methods employed and the equipment in use. In proper sequence the ages of a well are dealt with, from the flowing period, and the control of flowing wells, and the various pressure-recording devices required.

As the latent energy within the oil deposits is gradually expended, secondary methods of recovery are called for, and, in the chapter termed by the author "Pneumatic Pumping of Oil Wells," various forms of gas lift, multi-stage gas lifting, intermittent flow devices, displacement pumping, and plunger lift are discussed and described, together with the surface equipment utilized.

Some 135 pages are allocated to the various mechanical devices for lifting oil from wells, following decline of natural and induced methods of oil production. The fullest description is given of the types of equipment employed both in the wells and on the surface.

Discussion is next made of the methods employed on the field for improvement of recovery, efficiency, and reduction of production costs. The author emphasizes the importance of sand control in well operations and describes the various types of screens used in finishing the wells, which are also illustrated. Gravel packing or screening on the outside of the oil string, removal of inorganic precipitates from the well, which tend to prevent access of the oil, and flooding of the oil sands with oil from inside the tubing via the screen perforations, are surveyed. The author, by means of a diagram, emphasizes the advantage to be gained by completing wells with as large a diameter as possible, to ensure an increased influence over a larger drainage area, to allow of wider spacing of wells and consequent lower development and producing costs. This leads him to an explanation of the use of explosives for the purpose of increasing drainage area around a well, and also the treatment by acid (hydrochloric) to increase production from wells producing from limestone reservoir rocks and prevention of corrosion of casing by use of an inhibitor.

Having explained the most efficient methods of well operation and control, by use of latent energy within the oil deposits, also the various artificial methods for prolonging the flow life of wells, the author proceeds to the study of " Secondary Methods of Oil Recovery," in which are included the application of vacuum to wells, gas injection, water flooding processes, and, finally, various types of mining for oil for further recovery. A cross-section of the mine at Pechelbron is included, and an explanation of the later method employed at the mine, both for better recovery and greater protection.

The concluding chapters, under a heading " Preliminary Refining of Petroleum," deal with the treatment of oil on the field prior to despatch. This includes separation of gas from the crude, water dehydration methods, and breaking up of emulsion, so that when the oil is finally despatched it is as free from impurities as possible.

There is an excellent chapter covering the gathering of oil on the surface and local storage requirements, gauging, sampling, testing of oil and gas, followed by another chapter on bulk storage, transport by way of oil and gas lines with suitable tables for flow, resistance to flow, etc., also transport by road, rail, and water. In the final chapters the author deals with auxiliary plant, such as workshops, etc., and various departments and their personnel, and concludes with a detailed account of office methods and records as customarily applied to the industry.

The book is neatly bound and printed on good paper. Sufficient tables and diagrams with formulæ are provided to expedite the sundry arguments requiring elucidation.

Each chapter opens with a reasoned preface of the subjects embodied and, for those readers requiring still further information regarding any special subject in which they may be more particularly interested, a selective bibliography has been included at the end of each chapter.

The author has made this highly technical subject interesting reading, and the complete work forms a standard reference for all engaged in the industry.

This is a masterly treatise and no one is better fitted or more qualified than the author to undertake so gigantic a task. R. J. Ward.

IIme Congrès Mondial du Pétrole; Comptes Rendus. Published at 85, Boulevard Montparnasse, Paris, 6e. 11″ × 8⅝″. 5 volumes:

Vol. I. Geology, Geophysics, Drilling ; 1021 pp.; 36*s*.; $9.00.
Vol. II. Physics, Chemistry, Refining ; 1230 pp.; 60*s*.; $15.00.
Vol. III. Materials and Construction ; Utilization ; 902 pp.; 44*s*.; $11.00.
Vol. IV. Economics and Statistics ; 805 pp. 28*s*.; $7.00.
Vol. V. General Information ; 94 pp. 8*s*.; $2.00.

Prices given are for copies bound in cloth. Vols. I–V when purchased together are £7 9*s*.; $37.00. Postage and packing charges outside France : 10 per cent. extra on the above prices.

Publication of the *Proceedings* of the Second World Petroleum Congress, organized by the Association Française des Techniciens du Pétrole and held in Paris in June 1937, was completed early in 1939. There are five volumes. The contents of these volumes are approximately indicated by their titles given above. This list of titles does not do justice, however, to the remarkable range of subjects covered in each volume—a range far wider than was attempted at the First World Petroleum Congress of 1933. Altogether, there are 426 Papers, which, together with reporters' summaries, discussions, speeches at the plenary sessions, etc., make a total of over 4000 pages. It is a testimony to the vigour of the petroleum industry that the scientific studies should mainly relate to the three years intervening between the First and Second Congresses; and to the catholicity of outlook of the French Organizing Committee that nothing pertaining to petroleum was considered alien from the interests of the

Congress, whether legal, fiscal, archæological, statistical, educational or purely scientific in character.

Volume I comprises two parts. Approximately one-third of the book consists of general reports on petroleum developments in various countries between 1933 and 1936. The remaining two-thirds consists of Papers on geology, geophysics, drilling and exploitation respectively. The general reports were an innovation. Mainly historical in character, they form a valuable source of reference to the countries concerned. The geological Papers are for the most part regional studies. Geophysics is represented by twenty-three Papers notable for examples of specific surveys. Drilling is mainly concerned with rotary developments and nine Papers on drilling muds. The section on exploitation, *i.e.* production engineering, is somewhat disappointing. There are many aspects of reservoir dynamics not represented.

Volume II, the largest of the five volumes, contains Papers coming within the broad categories of " Physics, Chemistry and Refining " of petroleum products, although it is not always clear why some Papers (on lubricants in particular) were allocated to Volume II and others to Volume III (Section " Utilization "). The influence of chemical trends between 1933 and 1936 is clearly indicated by the sectional arrangement adopted—de-waxing, solvent refining, hydrogenation, utilization of petroleum gases, oxidation of lubricating oils. Additional sections deal with analytical methods and viscosity.

The first part of Volume III, " Materials and Construction," constitutes a grouping of Papers that is unique, in that it brings together the chemical engineer, the electrical engineer and the metallurgist to the study of problems encountered in refinery construction. The principal topics discussed are corrosion, high-pressure plant, evaporation losses and electrical equipment for refineries. " Utilization " in the second part of Volume III is mainly concerned with fuels and lubricants for internal-combustion engines.

Volume IV and the section which it represents, " Economics and Statistics," was admittedly an experiment—and a remarkably interesting and successful experiment it has proved to be. The title of the volume is somewhat of an omnibus character, because this volume treats of a wide diversity of subjects. There is, for example, a 150-page dictionary of petroleum terms in French, English, and German (in English alphabetical order). The preparation of this dictionary represents many months of compilation and checking by those responsible. There is a section on the archæology of petroleum; a section on legal enactments controlling exploitation in various countries; various Papers on standardization of methods of testing and nomenclature; and a short section on education.

The final volume of the *Proceedings* contains reports of speeches at various functions, the lecture by Prof. Henri Weiss, delivered before M. Albert Lebrun, President of the French Republic, and a list of the personnel of the different national committees, delegates, and members attending the Congress. A number of pencil sketches, reproduced from *La Revue Petrolifère*, add to the interest of this volume.

Three official languages were adopted—French, English, and German. It would probably be a fair approximation to say that 80 per cent. of the text is in French, 10 per cent. in English and 10 per cent. in German. Abstracts of the more important technical Papers have already appeared in the *Journal* Abstracts during 1939.

The President of the First Congress, in declaring open the Second, said that Research and Fellowship were the twin motives that underlay international scientific gatherings of such a nature. The research is recorded in these *Proceedings*. In due course it becomes absorbed into the ordinary workaday technique. The fellowship remains.

S. J. ASTBURY.

BOOKS AND PUBLICATIONS RECEIVED.

Petroleum Facts and Figures. 6th Edition. 1939. The American Petroleum Institute, New York, pp 190. $1.00.

The sixth edition of these statistical tables relating to the American petroleum industry continues the data of previous editions down to the end of 1938. The tables are grouped under the headings of Utilization, Production, Refining, Transportation, Marketing, Prices and Taxation, and General (Employment, Accident Rates, Fire Losses, etc.). The source of the data is indicated in the case of every table. Salient features are emphasized in the titles given to the tables (*e.g.*, " One Gasoline Pump in Use for every Twenty-one Cars," " Two-thirds of Wells Drilled find Oil," " California Largest Residual Fuel Oil Producer," etc.) and by an extensive use of " pictograms." A valuable Appendix contains particulars of the organization and personnel of the American Petroleum Institute, a directory of 121 petroleum associations in U.S.A., and a useful list of A.P.I. publications.

The book is sponsored by the Department of Public Relations of the American Petroleum Institute, and the Foreword pays a tribute to Mr. Edwin W. Esmay for the work of compilation and editing.

The Machining of Copper and Its Alloys. C.D.A. Publication No. 34. Pp. 108. Issued by the Copper Development Association, Thames House, Millbank, London, S.W.1.

This publication, which discusses the machining properties of copper alloys and summarizes modern machining practice as applied to these materials, contains a few pages on cutting fluids. These include alkaline solutions, and soluble, mineral, fatty, blended and sulphurized oils.

NEW PUBLICATION.

Petroleum. Vol. I, No. 1, October 1939. Size $8\frac{1}{2} \times 5\frac{1}{2}$ in. Pp. 1–36. Published monthly by Leonard Hill, Ltd., at 17, Stratford Place, London, W.1. Annual Subscription 10*s.* post free; abroad 12*s.* 6*d.*

The first issue of this publication contains the following signed articles : " Safety a Science," by C. L. Hightower ; " Petroleum Products in Industry," by S. F. Daley, and " Unit Processes in Oil Refining," by M. E. Clark. In addition, there are anonymous articles on pipe-line, underground storage, Germany's oil needs, and diesel fuel, together with editorial comments and items of topical interest. A selected list of recent patents is given.

INSTITUTE NOTES.

December, 1939.

FORTHCOMING MEETINGS.

Thursday, 25th January, 1940 : Northern Branch ; Luncheon Address by the President in Manchester. (Full details to be announced later by the Hon. Secretary of the Northern Branch.)

Friday, 26th January, 1940 : at 2.40 p.m. at the Royal Society of Arts, John Street, London, W.C.2.

Presidential Address by Professor A. W. Nash, M.Sc., F.Inst.Pet.

STUDENTS' MEDAL AND PRIZE.

The Council offers annually a Medal and Prize (to the value of Five Pounds in books) for the most meritorious essay on a subject connected with petroleum technology submitted by a Student Member of the Institute.

The conditions of award for 1939–1940 are on a different basis than in previous years, in that Students must submit essays on one of the subjects in the following list :

1. Geological structures favourable to the accumulation of oil.
2. A comparison of the various geophysical methods of prospecting for oil.
3. The controlling factors of deep drilling.
4. Mud fluids for drilling purposes.
5. The principles of three-dimensional flow of fluids through porous media.
6. Special products manufactured from petroleum.
7. The sweating of paraffin-wax scales.
8. The behaviour of fuel oils in compression-ignition engines.

Entries must be received by the Secretary of the Institute not later than 1st May, 1940.

No maximum length of essay is specified, but in general essays should be from 4000 to 6000 words in length.

Entries must be typed, and one carbon copy must be furnished in addition to the original copy. The Council is prepared to assist Students who experience difficulty in getting their MSS. typed.

BURGESS PRIZE.

Entries are also invited for the Burgess Prize (of value up to Five Pounds in books) for an essay dealing with some aspect of the economics of the petroleum industry, including the transport and distribution of petroleum products.

The conditions of entry for the Burgess Prize are the same as for the Students' Medal and Prize.

The Council reserves the right to withhold the award of either of these prizes, or to award prizes of such lesser values as it may decide.

ARTHUR W. EASTLAKE,
Honorary Secretary.

ABSTRACTS.

Loan of Periodicals.

Commencing with the November issue of the *Journal* (No. 193), every Abstract of which the original article is available at the Institute's office in Birmingham is indicated in the *Journal*. The original can be borrowed by members in Great Britain through the post.

Periodicals will be loaned for one week only. Members should send stamps to cover the outward postage. A reply-paid label will be issued for the return postage.

Photostat Copies.

Members either in Great Britain or abroad can be supplied with a photostat copy of any article marked in the Abstracts as so available. The charge for photostat copies is 1*s*. 3*d*. per page photographed. Orders should be accompanied by remittance.

JOURNALS WANTED TO PURCHASE.

The Institute is prepared to purchase copies of the following *Journals* at 5*s*. per copy, providing they are in good condition: Nos. 171 to 174 inclusive (January, February, March and April, 1938).

Copies for disposal should be sent to the Secretary.

BINDING OF VOL. 25 (1939).

Orders for binding Vol. 25 of the *Journal* may be forwarded to Richard Clay & Company, Ltd., 16 New Street Square, London, E.C.4, and should be accompanied by the separate parts and appropriate remittance.

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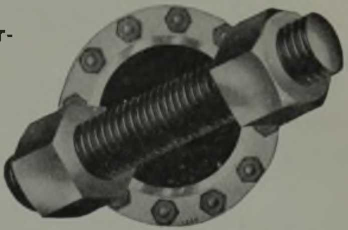

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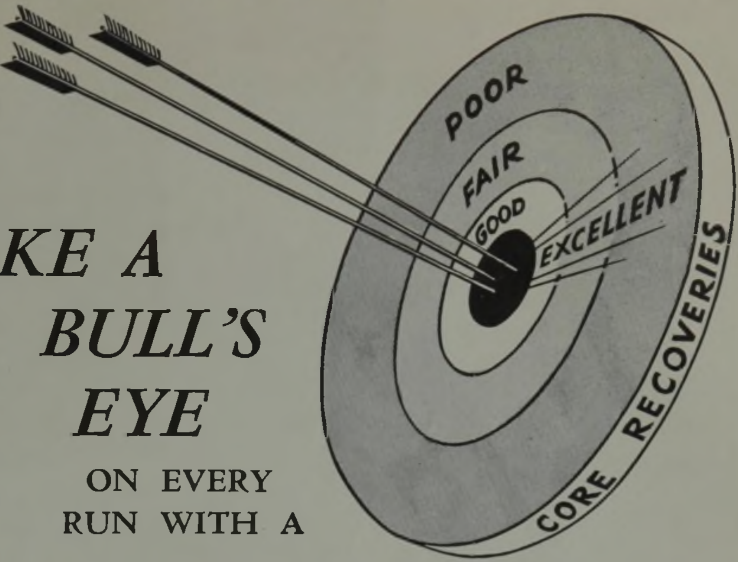
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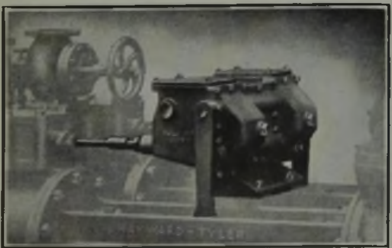

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