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PRESIDENTIAL ADDRESS.

THE DEVELOPMENT OF PETROLEUM REFINING.*

By PROFESSOR A. W. NASH, M.Sc.

THE purpose of this address is to record briefly, and in historical sequence, those developments in petroleum refining which have been of the greatest importance, in so far as they have led to present-day practice, bearing in mind that time and space do not permit of a comprehensive review of all phases of the subject.

At one time the sole activity of the refiner was that of distillation, and so little has the principle changed that even to-day distillation is the major function of the art of refining.

Distillation processes have been used by the earliest experimentalists, and Pliny (A.D. 23-79) describes a method of obtaining rosin oil by the application of heat followed by a primitive method of condensation. The Alexandrians are reported as having prepared oil of turpentine by distilling pine resin, whilst improvements in condensation apparatus are attributed to the Arabians, who are said to have been responsible for the discovery and extraction of a number of essential oils, and of alcohols from wine, by distillation methods.

In the work of Zosimus (*circa* A.D. 400) are described and illustrated certain types of distillation utensils, based upon apparatus which he saw in use in the Temple of Serapis at Memphis, one of the chief alchemical laboratories of that time.

Although the art of distillation had been known for several centuries, it would appear that alcohol distillation was not practised on any large scale until the eleventh century, when the alcohol industry as then established became the pioneer of all the distillation processes. Subsequently distillation came into use not only in the petroleum industry, but also in many other industries.

DISTILLATION.

The general principles involved in distillation are, like most principles in science, very simple in outline, but most complicated in detail.

* Address delivered at a meeting of the Institute of Petroleum held in London on Friday, 26th January, 1940.

Petroleum is a very complex substance, and in its conversion into vapour and its subsequent condensation into liquid the known scientific principles underlying the separation of simple mixtures cannot be applied with any high degree of accuracy to a process which is undoubtedly nothing more than an art.

Nevertheless, the distillation of petroleum, and its fractionation into the products then marketed, were carried out quite successfully in the earlier days, although it is doubtful if many refiners appreciated the part played in such processes by the hypotheses of Dalton (1801) and of Avogadro (1811), or by the law of Raoult (1883).

The simplest distillation apparatus, so well known to the "moonshiner," consisted of three parts: the still, the condenser and the receiver. These three parts are just as much essentials in the more complicated designs of present-day plant, the only addition of major importance being that of the fractionating column.

In this latter respect we owe much to Wurtz, Linneman and le Bel, and to Henninger for their ideas on fractionation, whilst the credit for the development of reduced-pressure distillation is due to Dittmar and to Anschutz. To Liebig we owe the laboratory condenser, and the development of the reflux condenser is due to Kolbe and to Frankland.

The coal oil produced by James Young (1847) was found to be unsatisfactory when used as a lamp oil to replace whale oil, which was becoming increasingly scarce at that time, and this demand for an alternative source of supply led to a rapid development of petroleum refining for burning-oil purposes. This was followed by an increasing demand for lubricants much in excess of the supply of animal, vegetable, and fish oils for this purpose, with the consequent increase in the number of petroleum refineries.

The earliest stills used in the distillation of petroleum were replicas of those which were then used in the coal-oil industry. These were heavy cast-iron vessels, round in shape, with capacities ranging from 200 to 2000 gallons, the only instrument of control fitted being a thermometer.

Although horizontal cylindrical stills, of much larger capacity and made of thin wrought-iron plate, were introduced in 1860, it was not until 1885 that the continuous-shell-still method of distillation came into general use. At about the same time Leet pointed out the advantages of bubble-tower rectification as used in the alcohol industry and recommended its use in petroleum refining. Nevertheless it was not until thirty years later that his advice was accepted by the petroleum industry, after some ten years' experimentation with packed towers.

It is true that for some years Heckmann had been using, in Europe, a column based on this principle for the redistillation of benzene from shell stills, reflux being obtained by means of a partial condenser, but such a principle was not in general use for many years after.

In the year 1905, or thereabouts, a number of packed vertical towers, stone being at first used as the filling, followed by baffle-plates, were fitted with partial condensers and used in the United States by the Standard Oil group, but it was not until difficulty was experienced in dehydrating the heavy California crude oil in shell stills that pipe-stills were first tried out for this purpose in the petroleum industry, although they had been used in coal-tar distillation as far back as 1890. These pipe-stills soon came into general use largely due to the efforts of Trumble, Fuqua, and of Bell. By 1911 pipe-stills and dephlegmating towers were used almost throughout California, and gradually spread to other fields in the United States.

Their development outside of America hung fire for some years, but as a result of an attempt in 1920 to standardize specifications of petroleum products, particularly those of gasolines, kerosines, and certain lubricating oils, the problems of the plant designer were very much simplified. The industry is now in possession of fractionating equipment of such flexibility and accuracy as has resulted in a remarkable reduction in the cost of manufacture of refined products, thus enabling the consumer to purchase at a price, until recently, unprecedented in the history of petroleum.

In considering the development of pipe-stills, of particular interest was the Trumble plant first used in 1911. The original large tubes were subsequently reduced considerably in diameter when the importance of radiant heat was appreciated by engineers responsible for furnace design. Thus the present-day multi-pass tubular stills, with their accurately machined return bends, may be said to owe their origin to Trumble. The advantages of the baffle-plate air-cooled dephlegmator and the heat exchanger, the former reducing the amount of re-running necessary and the latter effecting a considerable saving in cooling water and fuel costs, soon became evident. Great improvements have since taken place in regard to condensers: from the old worm condenser, containing coils of comparatively large-diameter pipe submerged in water, we now have nests of small tubes with multi-pass arrangements, which make for efficient heat transfer, together with floating heads, which allow for expansion, thus eliminating leakages.

An indication of the advance made in the distillation process of petroleum refining is best measured by a comparison of heat transfer then and now. In the radiant heat section of modern pipe-stills,

35,000 B.Th.U. per square foot projected area per hour are possible when necessary, as compared with 4000 B.Th.U. maximum in the old shell stills.

The reduction in the quantity of oil being heated as the result of the introduction of pipe-stills necessitated much closer control of operating conditions, with the result that automatic devices for recording and controlling temperatures and liquid levels were introduced. These devices have now reached a high standard of precision, largely due to the practice of cracking, which was established about the year 1920, the four most important processes then in existence being those of Burton, Dubbs, Cross, and Greenstreet.

The last three processes made full use of the knowledge which had then been acquired regarding the value of pipe-stills, baffle-plate towers, and heat exchangers, to be followed by the use of the bubble-towers introduced as the result of experience in casing head gasoline extraction. Thus forty years after Leet's attempt to popularize the bubble-tower in petroleum refining, it became firmly established in 1925.

Although advances have been perhaps greater in the design of pipe-stills than in any other branch of petroleum distillation, nevertheless the most important improvements which have taken place are undoubtedly in connection with fractionating towers, probably due to the longer experience of the distillers of alcohol. The present-day multiple side-stream fractionating tower, really a combination of several fractionating towers in one unit embracing vapour heat exchangers in connection with side strippers, the closeness of fractionation of which depends so largely on adequate reflux ratio, has resulted in a marked increase in thermal efficiency and in the cleaner separation of fractions. Indeed, the gap between the end-point of one fraction and the initial boiling point of the next shows that the modern bubble-tower can give much closer separation than the standard Engler flask, a fact which would have been hard to realize in 1926, when these towers were first installed on a commercial scale.

CRACKING.

As a result of the developments just referred to, the art of cracking benefited to an even greater extent, and to-day it may be said that distillation and cracking plants are so flexible as to be capable of handling practically every type of crude oil or distillate, from the heaviest oils to the lightest spirits, or even gases.

The cracking process has contributed more towards the conservation of crude oil than any development in the history of the petroleum industry, for to have produced all motor-fuel requirements from

straight-run gasoline would have necessitated the raising of enormous quantities of crude oil, with the subsequent production of other fractions, far in excess of requirements. This would have led to such waste as to have upset the economic efficiency of the petroleum industry of the world, because to-day nearly half of the gasoline consumed is produced by cracking.

The earlier experimental work on pyrolysis by Faraday (1830) and by Berthelot (1860) merits record, whilst Young (1865) and Dewar and Redwood (1889) were undoubtedly pioneers of the present highly efficient methods of cracking which are capable of converting the most unpromising crude oil and heavy and medium residues into gasolines of much higher grade, judged by the present-day standards, than most of those obtained by straight run distillation. In paying such tribute the names of Burton, Ramage, Greenstreet, Hall, Rittman, and MacAfee should also be mentioned.

Able to profit from the use of pipe-stills which had then become established in distillation practice, the designers of those original plants which have since acquired commercial success at first based their plans on empiricism and the technology of cracking acquired by experience. A study of the thermodynamics and chemistry of cracking has helped considerably in improving the design of these plants, and a better understanding of time and temperature relationship led to the knowledge of the advantage of separating the heating and reacting zones, which resulted in the rapid development of such processes as the Cross, Dubbs, Tube and Tank, Jenkins, and the Holmes-Manley.

The increasing demand for gasolines of higher anti-knocking qualities was a cause of the return to the so-called vapour-phase methods of cracking, such as the modified Gyro process, that of De Florez and the True Vapour-Phase process, but Midgley's discovery (1923) of the value of lead tetraethyl as an anti-knock dope resulted in a check to the further development of vapour-phase cracking, as such, for a short time.

With the higher cracking temperatures now in use, it is impossible to distinguish between liquid- and vapour-phase conditions, but if the present demand for high-octane fuels continues, it is probable that straight-run spirits will not have a ready sale in the future, unless they have been thermally treated previously. It is for this reason that the process, known as reforming, has advanced to such an important position in refining to-day, and the gases formed by this and vapour-phase cracking are now proving of extreme value as a source of raw material for chemical synthesis, a subject which will be discussed later.

Simultaneously with these developments experiments were being carried out to produce heavier oils in the lubricating range without destroying their lubricating properties as the result of decomposition, and so 1930 saw the introduction of the low-absolute-pressure cylindrical-still units of Schultz and of Steinschneider, which were later converted into continuous multiple still operations. The principle of these units has since been adopted in pipe-still and bubble-tower operations, although not necessarily confined to the production of lubricants only.

The next aim of the plant designer was to combine the distillation and cracking processes in one unit, which culminated in the first of the very large modern combination units being erected in 1932 for the Standard Oil Company of Indiana.

To-day combination units are running with a throughput as high as $1\frac{1}{2}$ million gallons a day, which include as an integral part of their process, crude topping, primary cracking, vapour-phase cracking, fuel-oil flashing, reforming and vapour-phase refinement resulting in a very high rate of heat conservation and the maximum production of gasoline of comparatively high octane value.

In this latter connection the future possibilities of catalytic cracking should here be considered.

Whilst catalytic cracking was known in the earlier days of experimental pyrolysis, it has but recently been employed in commercial practice on a large scale, and units are now in operation with throughputs as high as 600,000 gallons daily.

Some of the chief claims of the Houdry method are liquid recoveries as high as 99 per cent., with a corresponding decrease in gas and coke production yielding gasoline of 80 C.F.R.M.M. octane numbers from any type of charging stock heavier than gasoline, suitable as base stock for aviation fuels.

Mention should here be made of a method of catalytic reforming known as cyclization, which, as its name implies, can convert hexane into benzene, heptane into toluene, etc., and is a much more valuable source of aromatics than the older method of gas pyrolysis so well known to us through the researches of the late Professor R. V. Wheeler.

Where products other than gasoline are of importance there are available the combination of high-vacuum pipe-still distillation, bubble-tower fractionation aided by flash jugs, efficient heat-exchanging devices, and modern instrument control of such a high degree of efficiency as to enable the refiner to produce a more uniform product at a much lower cost than previously from a wider range of raw materials. This has been reflected in the cheapness with which the

consumer can purchase his requirements despite the burden of taxation which the products of petroleum have had to bear in this country.

REFINING.

It has been stated that the progress of chemical science throughout the ages can best be measured by the quantity of sulphuric acid used throughout the civilized world, and this statement applies no less to the petroleum industry.

One of the earliest uses to which petroleum was applied was that of illumination, and as a result of Scottish shale-oil practice, which originated with Young in 1847, sulphuric-acid treatment followed by caustic-soda washing became universal throughout the industry; in fact this process is still the most common method to-day for the removal of undesirable components from burning oil and other distillates, particularly cracked spirits.

It was not until 1884 that lubricating oils were first produced on a large scale as overhead products, an improvement ascribed to Samuel Downer, who was also responsible for the chilling of oils followed by the pressing of wax as carried out by earlier manufacturers on exactly the same principle as that employed at the present time.

Exactly when the adsorptive properties in filtration were first utilized by our industry cannot be traced, but the phenomenon of adsorption was reported by Scheele in 1773, and bone black had been used for filtering for centuries before the advent of Florida earth in the petroleum industry about the year 1898. By 1906, however, Florida earth was in common use by manufacturers of lubricating oils, and at the same time sweating was introduced for the manufacture of wax of varying degrees of hardness, an operation which was substantially the same then as at the present time.

Some few years after this there arose a demand for lubricants for petrol engines, a demand which has been growing ever since, and the contact method of filtration for the stabilization of acid-treated oils was investigated by Gray; a study which undoubtedly led to his method of vapour-phase treatment of cracked spirits.

In 1885 Frasch was responsible for a process of refining in which kerosine was distilled over black copper oxide, a discovery which was followed by several processes for the removal of mercaptans by forming their metallic derivatives.

Cold settling was discovered quite by accident in 1890, and in 1894 the present low-cold-test and bright-stock development had become firmly established. This was followed in 1920 by centrifuging,

which opened up a new source of supply of bright stocks from crude oils other than of Pennsylvania origin. To-day, however, both crystalline and amorphous types of wax, and mixtures of both, are successfully extracted by the solvent dewaxing process, using either single-component solvents or two-component solvents, and it is probable that most dewaxing installations will be of the solvent type in the near future.

Of the so-called physical methods of refining the use of bauxite was amongst the earliest, and this method was originated by the Burmah Oil Company at their Syriam Refinery in 1910, whilst the first commercial solvent-extraction process using liquid sulphur dioxide was introduced by Edeleanu at about the same time.

This was followed by various methods for the removal of mercaptans, thioethers, and disulphides in petrol by oxidation employing the so-called sweetening process using hypochlorite in alkaline solution, sodium plumbite, and copper salts, etc.

It would appear also as though the position hitherto occupied by sulphuric acid in the refining world will now be affected by the new solvent-treating method of refining, particularly in the case of lubricating oils. As in the case of wax removal, there are both single- and double-solvent processes.

Of the double-solvent type, sulphur dioxide and benzol, and also cresylic acid and propane, are in common use, whilst a number of plants are now in existence using such single solvents as furfural, phenol, liquid propane, $\beta\beta$ -dichlorethyl ether (chlorex), and nitrobenzene.

The solvent treatment of oils has re-introduced the old problem of oxidation and consequent acid formation. This has resulted in a great deal of study into the inhibiting properties of certain compounds. Nevertheless, the improvements obtained in the sludging and carbonizing properties of lubricating oils, and also in their viscosity temperature relationships, more than offset any increase in the cost of manufacture as the result of having to add to them oxidation inhibitors.

Although the advances in refining methods have not been quite so spectacular as on the distillation side, largely due to the fact that experimental work in refining is usually carried out on a smaller scale in laboratories, whereas advances in distillation have evolved as the result of experiment on a more technological basis, nevertheless they have been equally as important, and have contributed just as much to the improvement of the product, as well as the lowering of the cost of such product.

In all experimental work of this kind the petroleum chemist is

handicapped by his lack of knowledge concerning the chemical constitution of the product he is attempting to improve, for whilst it is true that the chemistry of the constituents of the lower-boiling fractions of petroleum is now fairly well established, that of the higher-boiling fractions, particularly in the lubricating oil range, is practically unknown.

SYNTHESIS.

No industry, the operations of which are based on the application of scientific principles, can be said to be developing to the fullest extent possible unless a study of synthesis is an important part of its research programme. In this respect the petroleum industry has certainly not been backward, as witness the development of the so-called "poly" plants, to quote only one example.

Although it has long been known that unsaturated hydrocarbons, no matter whether in the lower- or the higher-boiling fractions, are readily polymerized, their use on a commercial scale for this purpose is a comparatively recent development.

The production of high anti-knock spirit and the formation of aromatic liquids of even higher molecular weight from the lower olefines containing from two up to four carbon atoms in the molecule should here be mentioned.

The synthesis of lubricating oils, not only by polymerization, but also by means of the Friedel and Craft reaction, in which cyclic and chlorinated straight-chain hydrocarbons are combined in the presence of aluminium chloride is a recent development of some note. In fact during the last few years much research has been devoted towards the building up of new molecular structures.

It was not until some twenty years ago that the fundamental chemistry of the hydrocarbons awakened much interest in the petroleum industry, although in a few isolated cases investigations into this subject had been made by de la Rue, Markownikoff, Mabery, Sabatier, and Kishner.

More recently a considerable amount of work has been carried out by our contemporaries, and it would be remiss of me not to express the gratitude of the members of this Institute to them and to the larger oil companies for the public-spirited manner in which they have made themselves responsible for the bulk of this work, which has been carried out not only in their own laboratories, but elsewhere, and also for allowing the results of these investigations to be published freely, very largely through the medium of our own *Journal*.

It is probable that the increased demand, caused by the last war,

for more suitable solvents for the lacquer industry really provided the stimulus for the production of chemical intermediates and special products from petroleum, although it should be recalled that the Russian petroleum industry had been utilizing sulphonated acid sludge and naphthenic acids on an industrial scale long before 1914.

Furthermore, the development of cracking led to the production of enormous quantities of gases containing varying amounts of olefines for which a use had to be found.

In 1862 a small quantity of alcohol made from coal-gas ethylene by means of the sulphuric-acid process was exhibited at the London Exhibition, but the commercial preparation of ethyl alcohol was, until recently, entirely from molasses.

By 1920 the production of *isopropyl* alcohol from cracking gas was established on a large scale, and this was quickly followed by the preparation of ethylene glycol as an anti-freeze agent, together with many derivatives of the glycols. The chlorination of natural gasoline was the next step in development, and by 1926 the preparation of amyl alcohol and acetate had taken form. Similarly secondary butyl alcohol is being produced from butene, whilst its acetate has proved of value as a lacquer solvent.

More recently, because of the fact that propylene appears in greater quantity than *isobutylene* in cracked gases, it was thought advisable to convert it into *diisopropyl* ether by the action of sulphuric acid in order to meet the ever-increasing demand for high anti-knock fuel.

This ether has a slightly higher blending octane value than commercial *iso*-octane, and for a given tetraethyl lead concentration in corresponding blends it is also superior. Its miscibility with water and relatively low calorific value are, however, disadvantages. The latter is specially important as it is 18–20 per cent. less than that of *iso*-octane, and when the ether is present to the extent of 40 per cent. in aviation fuel, the calorific value of the latter is some 7 per cent. lower than for a similar blend with *iso*-octane.

It should be realized, however, that *diisopropyl* ether is not intended to replace *iso*-octane, but rather to augment the supply of high-octane blending material.

Developments of outstanding importance during the last few years have been the combined pyrolysis and polymerization process, whereby high-grade gasolines are now produced synthetically from both natural and cracked hydrocarbon gases, the production of benzol by high-temperature cracking, and the preparation of *diisobutene* by the polymerization of *isobutene* in the presence of sulphuric acid.

As was to be expected, it was but a short step to the dehydrogenation of gaseous paraffins followed by the treatment of olefine-paraffin mixtures, as mentioned above. Ethane, propane, and *n*-butane can be handled to great advantage by dehydrogenation to the corresponding olefines. Of these, propylene and the butenes can be alkylated with *isobutane* or, alternatively, the butenes can be polymerized to dibutenes, which may be hydrogenated to octanes. Ethylene produced in a similar fashion may be polymerized to motor fuel. In fact, it may be stated that all the hydrocarbon constituents of natural gas may now be converted into aviation fuels with Octane Numbers (C.F.R.M.M.) of 90 and above, with the exception of methane and ethane. As these hydrocarbons are the most prolific in natural gas, it should be emphasized that their utilization is a matter of considerable urgency.

While ethane may be dehydrogenated to ethylene, which may be subsequently polymerized, the polymers produced do not yield high octane numbers on hydrogenation. Also ethylene has not yet been satisfactorily alkylated with *isobutane* in the presence of sulphuric acid on a commercial scale, but the Phillips Petroleum Company of America now effect this by thermal treatment under pressure, and in this manner produce neohexane (2 : 2-dimethylbutane).

The next step was to reduce the temperature and pressure of the polymerization processes, and catalysis has been successful in this, not only for the production of motor fuels and of lubricating oils, but also in suppressing secondary reactions whereby the polymerized olefines had a tendency to revert to hydrocarbons saturated in character.

The first mention of the polymerization of *isobutene* by means of sulphuric acid was attributed to Butlerov in 1876, and the method now in general use is effected by acid catalysts, either in the liquid phase—*e.g.*, sulphuric acid—or in the solid phase, such as orthophosphoric acid supported on a carrier, and considerable quantities of *diisobutene* are now being prepared in this manner.

In the stabilization of gasolines the *isobutane* fraction can be extracted, which, together with that found in cracking gases, represents a vast potential source of supply for the production of *iso*-octane.

The method of preparing *iso*-octane from *diisobutene* using nickel as a catalyst was first described by Edgar in 1926, but a more recent development has been the production of high-octane fuels by the hydrogenation of the condensation product from a mixture of *n*-butene and *isobutene*. In this manner one molecule of the polymer is formed from one molecule of *isobutene*, instead of from two molecules

as with diisobutene, thus practically doubling the yield of product for a given quantity of the branched hydrocarbon. The product on hydrogenation gives a fully saturated paraffinic fuel of 96 octane number, possessing a good lead response, and as there is always a greater proportion of normal butenes in cracked gases than isobutene this process is likely to take precedence.

It has been estimated that the hydrocarbon gases in the United States of America form a yearly potential supply of 9000 million gallons of polymer gasoline, including over 1000 million gallons of *iso*-octane fuel of 95-100 octane numbers. By applying these processes to waste refinery gases, an appreciable conservation of crude oil is accomplished.

The economics of these so-called "poly" plants are attractive, and appear to be of a sound profitable nature. It is no surprise therefore that polymerization plants now in operation or under construction will have an aggregate annual output exceeding 700 million Imperial gallons.

A point of interest at this stage is the number of small scale "made-to-measure" plants of the "midget" type which are being installed in many refineries.

To quote one such plant employed to process only a quarter of a million cubic feet of cracked distillate stabilizer tops daily, this yields 2800 Imperial gallons of 83-85 octane-number polymer gasoline per day. Using direct labour the plant cost £2000, and the capital cost was recovered in 36 days. The total output of the refinery was increased upon the introduction of this plant by 7 per cent.

The economic limit of polymerization plants is said to involve a feed rate of 280,000 Imperial gallons of liquid butanes and butenes giving 86,000 Imperial gallons of finished polymer gasoline per calendar day.

Rapid advance is taking place in producing any given commodity at smaller costs. Thus, according to Egloff, gasoline of 125 octane number, having 50 per cent. greater power output than present 100 octane gasolines, has been produced experimentally in petroleum laboratories. The cost per gallon of this fuel has been reduced by more than 98 per cent. due to experiments carried out over a period of one year—that is, from just over £720 to just over £10 a gallon. It is hoped that the cost may be reduced sufficiently in the near future to permit commercial production.

Although not essential for to-day's motor cars, the super-octane gasolines are a major requirement of the nation's flying forces. American refineries are now producing the world's only adequate supply of fighting-grade aviation gasoline.

It is perhaps of interest at this stage to mention that the tars

produced as by-products in a benzole pyrolysis process are themselves capable of producing good yields of 90-100 octane-number gasolines of very low freezing points by the destructive hydrogenation of the polynuclear aromatic molecules, which are the main constituents of these tars.

One cannot finish a review of these processes without discussing in greater detail those plants which use sulphuric acid as the polymerizing agent. One process of considerable importance is the polymerization of *isobutene* to *diisobutene* and then hydrogenating the dimer to *iso-octane*. The reaction is the well-known one which follows two steps: firstly absorption of *isobutene* with subsequent hydration to the tertiary alcohol, and secondly dehydration and polymerization. The concentration of the acid is of vital importance, and the time of contact is another important factor.

After absorption of *isobutene* by acid the alcohol-acid mixture is passed to a polymerization vessel and heated until polymerization is complete. When this mixture of polymers is hydrogenated over nickel, 2:2:4-trimethylpentane or the coveted *iso-octane* results.

In this respect the preparation of high-octane aviation fuel by the Sulphuric Acid Alkylation Process as put forward by A. E. Dunstan and his collaborators, as well as the Shell Group, should here be mentioned, and a thought given to the much-discussed work of Ormandy dealing with the action of strong sulphuric acid on olefines, first published in our *Journal* in 1927.

This alkylation process affects directly the union of *isoparaffins* with olefines in approximately equal volumes to form branched-chain paraffins in the presence of sulphuric acid as the catalyst and in a single-stage operation.

In this manner yields of *isoparaffins* of high-octane number are more than double those obtainable by the two-stage process of polymerization followed by hydrogenation already referred to.

Before leaving this subject, the question of isomerization should be considered. Isomerization reactions in the field of high-octane-number motor fuels have enormous potentialities.

As is well known, saturated hydrocarbons exist either as straight or branched-chain compounds, and as the latter have higher anti-knock qualities than the former in the gasoline range—an excellent example of the difference in their behaviour in an engine being *n*-heptane and *iso-octane*—considerable interest in the controlled changing of straight chains to branched chains is now being displayed by those interested in motor fuels.

To these, isomerization is important from three points of view at the moment:

- (a) Conversion of *n*-butane into *isobutane* for use in alkylation processes.
- (b) Conversion of *n*-pentane into *isopentane* for use in 100 octane number aviation fuels.
- (c) Increasing the octane number of gasolines generally.

With regard to the latter point the extension of controlled isomerisation into the broad gasoline boiling range is a problem which should receive immediate attention.

The credit for the discovery of isomerism is undoubtedly due to Dalton, who, by distilling fatty oils in 1820, prepared a gas which resembled olefiant gas but appeared to contain twice as much carbon and hydrogen in a given volume. In 1821 W. Henry prepared a similar constituent from coal gas; Faraday then isolated this constituent from the condensation products of oil gas and found that the compound contained carbon and hydrogen in the same proportion as in olefiant gas (ethylene), but had twice the density, confirming Dalton's suggestion that probably the molecules of the new gas consist of two of olefiant gas, thus giving us the first example of isomerism.

Prior to these later developments, however, an attempt had been made to produce high-octane fuels by means of cracking under more severe conditions than normally, which resulted in distillates being formed which deposited a higher proportion of gumming materials. These gumming hydrocarbons were found to be a valuable source of supply of material suitable for conversion into resins.

For this purpose certain oils are now cracked to produce distillates containing a high proportion of olefines and diolefines, both cyclic and straight chain and aromatic and substituted aromatic hydrocarbons, solely for the manufacture of synthetic resins of different colour and hardness, by means of polymerization in the presence of aluminium chloride. Such resins can be used in varnishes or converted into moulded products.

Solvents derived from petroleum have long been used in the perfume and oil-bearing-seed industries, in bone degreasing, and in the dehydration of alcohol. They have also been used as cellulose lacquer diluents, in the manufacture of printing inks, as thinners in paints and varnishes, and in various polishes, as well as for many purposes too numerous to mention.

Solvents may be derived from both the paraffin and the olefine hydrocarbons. From the saturated hydrocarbons are obtained the chloroparaffins such as methyl chloride, methylene dichloride, chloroform, and carbon tetrachloride, as well as the amyl alcohols and their

esters referred to earlier. The olefines serve as raw materials for other chlorohydrocarbons and ethers, also acetone and its homologous ketones and their products. This is no recent discovery, however, as the power of hydrocarbons with double bonds to react additively with the halogen elements was known as far back as 1795.

The chlorinated products of petroleum are also playing an important part in the preparation of extreme pressure lubricants, and the Friedel and Craft reaction, referred to earlier, for the production of synthetic lubricating oils having high viscosity index properties, from chlorine and wax and heavier oils, may assume some importance in the future, as has been the case with the specialized product known as Paraflow.

Natural gas has been playing its part in other directions, notably as a source of hydrogen and dry ice, as a result of its reaction with steam. There is no doubt, however, that alcohol production is likely to prove one of the most important industrial outlets for the utilization of natural and olefine gases and in the production of pure chemical compounds in the future.

The relative cheapness of chlorine, together with the abundance of cracking gas within recent years, have led to the production of ethylene dichloride for solvent purposes and as an important material for organic synthesis. One of its more recent important uses has been its utilization with ethylene dibromide to cause more complete expulsion of the lead from the engine after combustion when ethyl fluid is used for suppressing knock in motor spirits.

In discussing the use of chlorine, the work of E. C. Williams on the synthesis of glycerol from petroleum gases should here be considered.

The fact that trichloropropane was difficult to manufacture economically by the direct chlorination of propane, or by the further chlorination of 1 : 2-dichloropropane, has long been recognized, and therefore the hydrolysis of 1 : 2 : 3-trichloropropane was considered out of the question.

However, the direct substitution of chlorine into the propylene molecule in such a way that the unsaturated bond remains unaffected has led to a method of synthesis of allyl chloride, which may then be transformed to glycerol by three alternative processes: either conversion to 1 : 2 : 3-trichloropropane by the addition of chlorine, or to dichlorohydrin by chlorohydrination, or by preliminary hydrolysis to allyl alcohol followed by chlorohydrination to glycerol monochlorohydrin.

The production of synthetic rubber from such materials as 1 : 3-butadiene prepared from the dichloride of butane, and isoprene prepared from the dichloride of *isopentane*, is also once more receiving attention.

Despite the advances, of necessity so briefly recorded in this address, the production of chemical intermediates from petroleum is but still in its infancy, and it is only by organized research, not necessarily with a view to immediate financial gain, that we can hope to advance our knowledge in this respect.

It should also be borne in mind, to quote only one case, that the method of manufacture whereby the cost of *iso*-octane was reduced from £5 to well below 2s. a gallon has been perfected within the last three years. Triptane (2 : 2 : 3-trimethyl butane), which is believed to have anti-knock properties even better than those of neo-hexane, and has so far been prepared by the hydrogenation of the olefine formed by the dehydration of 2 : 2 : 3-trimethyl butanol-3, is a discovery which has been made only within this last twelve months.

Petroleum consists essentially of compounds formed from two elements only, carbon and hydrogen. Carbon is the key element of organic nature, and any organic compounds capable of being synthesized in the laboratory should be capable also of being produced from petroleum. The value of research to the future of industry and to the industrial life of the nation cannot be over-estimated, and at the present time research is of greater necessity than ever, particularly when the nation is engaged in a scientific war. Such research should be of a wider scope than that of any of the Government Departments, the outlook of whose scientific staffs is often limited to their own special problems.

Research in petroleum science is being intensively carried out throughout the world, particularly in enemy territory where the whole of the scientifically trained men still remaining have been organized and are fully employed in professional work.

In this country, it is equally necessary that the whole of the scientific knowledge available, both of teams of laboratory workers and of individual scientific workers, should be fully employed, mainly on problems whose solution is necessary for ultimate victory over the enemy, but not neglecting the fundamental research essential to ensure that at the end of the war our industries are able to compete on favourable terms with all other countries.

To those who have done me the honour of reading this address I recommend these latter remarks for their earnest consideration.

SOLUBILITY OF CRACKED RESIDUES IN SOME SOLVENTS.

By J. VOSKULL

SYNOPSIS.

When cracked residue and fuel oil containing this product are diluted with different quantities of a precipitant hydrocarbon, and the percentages of precipitate are plotted against the dilution, "precipitation curves" are obtained. These lines are practically horizontal at high dilutions of aliphatic hydrocarbons and for *cyclohexane*. When adding large amounts of benzene to a cracked residue not containing sludge, a certain amount of precipitate is also formed. This precipitation curve is also horizontal at high dilutions. In the presence of sludge, small quantities of benzene may peptize part of the sludge, whereas larger quantities may again increase the amount of the precipitate; so a minimum in the curve is formed. This is illustrated by an example.

The results of these experiments were used to make a study of the effects when a cracked residue or fuel is extracted with pure benzene (for instance, in the Sediment-by-Extraction Test).

This led to the conclusion that the amount of the sediment after the extraction is dependent on the viscosity of the cracked residue or fuel and on the sizes of the pores in the extraction thimble.

In the second part of this article a comparison is made between the percentages benzene-insoluble in residues highly diluted with benzene and after a Soxhlet extraction of the pentane-insoluble with solvents increasing in solvent power up to benzene.

The extraction method gives higher values than the dilution method. It is thought that the constituents which stay in solution with even great quantities of benzene are precipitated with pentane and lose their solubility in benzene during the step-by-step extraction in which the adsorbed protective layers are removed.

The colloid-chemical nature of the cracked residue is clearly demonstrated by this.

INTRODUCTION.

In a previous publication¹ an investigation into the formation of precipitate in cracked residue and fuel oil containing this product, when increasing quantities of a precipitant hydrocarbon—*e.g.*, 60/80 gasoline were added—was reported. By plotting the quantity of precipitate as function of the dilution, "precipitation curves" were obtained. An extrapolation of these precipitation curves to zero dilution gave the quantity of "sludge"—*i.e.*, flocculated asphaltenes, etc.—which could not be kept in solution owing to the insufficient solvent power of the product.

In the above publication mention was also made of the application of gasoline-benzene mixtures to "flatten out" the precipitation curves so as to simplify the extrapolation.

The present publication describes the results of the continued investigation into the shape of the precipitation curves and the solubility of cracked residues in some solvents frequently applied in practice.

SOLVENTS USED.

In Table I the solvents are given, together with some physical properties. The solubility of Dubbs residue in these solvents increases from pentane to benzene in the order shown.

TABLE I.
Specific Gravity and Refraction of the Solvents Used.

Solvent.	Sp. gr. 20/4.	Refractive index, 20° C.	Details.
Pentane	0.6257	1.3575	Made from topped gasoline. Contains about 12% of isopentane.
60/80 Gasoline (I.P.T. Petroleum Spirit)	0.6847	1.3850	Mixture of <i>n</i> -hexane, <i>iso</i> heptane, and some naphthenes.
<i>cyclo</i> Hexane	0.7785	1.4263	Obtained from hydrogenated benzene chemically pure.
Carbon tetrachloride	1.595	1.4630	Commercial product; chemically pure.
Benzene	0.8755	1.4990	Chemically pure, free from thio-pheno.

PLOTING OF THE PRECIPITATION CURVES.

In theory the procedure is very simple: the residue is diluted with a certain volume of the precipitant liquid per gram of residue, and the resultant quantity of precipitate is then determined. In practice, however, great difficulties are encountered.

In the first place, the mixtures cannot be filtered, so that they have to be centrifuged. In his experiments the author used a series centrifuge from the International Equipment Co., Boston, U.S.A., with A.S.T.M., long-tapered bottom centrifuge tubes. These tubes were used in two sizes: 50 ml. for low dilutions and 100 ml. for high dilutions.

The centrifuge could develop a velocity of 3000 r.p.m. with the 50-ml. tubes and of 1800 r.p.m. with the 100-ml. tubes. The radius from the centre to the middle of the tube was 21 cm.

The centrifuging was continued until there was a distinct boundary line between precipitate and supernatant liquid, which did not shift in position on continued centrifuging. The precipitates were then washed out and rinsed with carbon disulphide into weighed gum dishes (I.P.T. G. 25 (T)). The percentage of precipitate was found by re-weighing the dishes after evaporation of the carbon disulphide and drying of the precipitate in an oven at 110° C.

Another difficulty was experienced in washing out, since the precipitate entrained an appreciable quantity of the medium, from which a fresh quantity of precipitate was formed when washing with the precipitant liquid. In the previous article¹ this wash-error was eliminated by using the so-called correction formula:

$$g = \frac{AE(1+x) - \frac{ETV}{100}}{E(1+x) + A - V - \frac{ET}{100}};$$

g = weight of precipitate to be determined;
 A = weight of precipitate to be obtained;
 E = weight of residue employed;
 x = degree of dilution (with a dilution 1 : 1, $x = 1$; for 1 : 2, $x = 2$, etc.);
 T = percentage of precipitate at very high dilution;
 V = volume of precipitate in centrifuge tube.

The correction formula is based on the assumption that the specific gravities of cracked residues and of precipitates are equal, or approximately equal, to 1. Generally this may be taken to be sufficiently correct, but with mixtures of residue and gas oil with a high proportion of gas oil this may not be the case, and the formula was accordingly modified so as to make allowance for the specific gravity.

When in the derivation of the formula the specific gravity of the residue or mixture (d_1) and that of the precipitate (d_2) are taken into account :

$$g = \frac{AE\left(\frac{1}{d_1} + x\right) - \frac{ETV}{100}}{E\left(\frac{1}{d_1} + x\right) + \frac{A}{d_2} - V - \frac{ET}{100 \cdot d_2}}$$

ACCURACY OF THE RESULTS.

It is of importance to know what value can be attached to the results obtained. The correction is the greatest at low dilutions or if the sludge can be centrifuged off at once because the residue or fuel oil has a sufficiently low viscosity (dilution 1 : 0).

It can be calculated that the absolute accuracy in the extrapolated sludge values is 3-4 units in the first decimal place. It follows from this that the accuracy is greater with high than with low sludge values.

PRECIPITATION CURVES WITH 60/80 GASOLINE, BENZENE AND MIXTURES OF THESE.

A mixture of a cracked residue A and a gas-oil with a sludge content of 3.4 per cent. was diluted with different percentages of 60/80 gasoline,

TABLE II.
Relation between the Percentage of Precipitate in the Residue A—Gas Oil Mixture and the Dilution with 60/80 Gasoline and Mixtures of this with Benzene.

Dilution.	60/80 gasoline.	90 gasoline, 10 benzene.	80 gasoline, 20 benzene.	70 gasoline, 30 benzene.	60 gasoline, 40 benzene.	50 gasoline, 50 benzene.	60 gasoline, 40 benzene.	30 gasoline, 70 benzene.	20 gasoline, 80 benzene.	10 gasoline, 90 benzene.	Benzene.
1 : 1	4.3	4.1	4.0	3.6	3.4	3.1	2.8	2.5	2.1	1.8	1.6
1 : 1	5.3	4.4	4.3	3.7	3.4	2.9	2.6	2.1	1.5	1.3	0.9
1 : 1	6.1	5.1	4.8	4.0	3.4	2.8	2.1	1.5	0.9	0.5	0.14
1 : 2	7.0	5.8	5.1	4.2	3.5	2.9	2.0	1.3	0.6	0.5	0.12
1 : 3	—	—	—	—	—	—	—	1.4	0.6	0.4	0.14
1 : 4	—	—	—	—	—	—	—	—	—	0.5	0.16
1 : 10	—	—	—	—	—	—	—	—	—	—	0.7
1 : 20	—	—	—	—	—	—	—	—	—	—	1.3
1 : 40	—	—	—	—	—	—	—	—	—	—	1.5
1 : 100	8.8	6.9	5.9	5.0	4.3	3.7	3.1	2.5	1.8	1.8	1.6

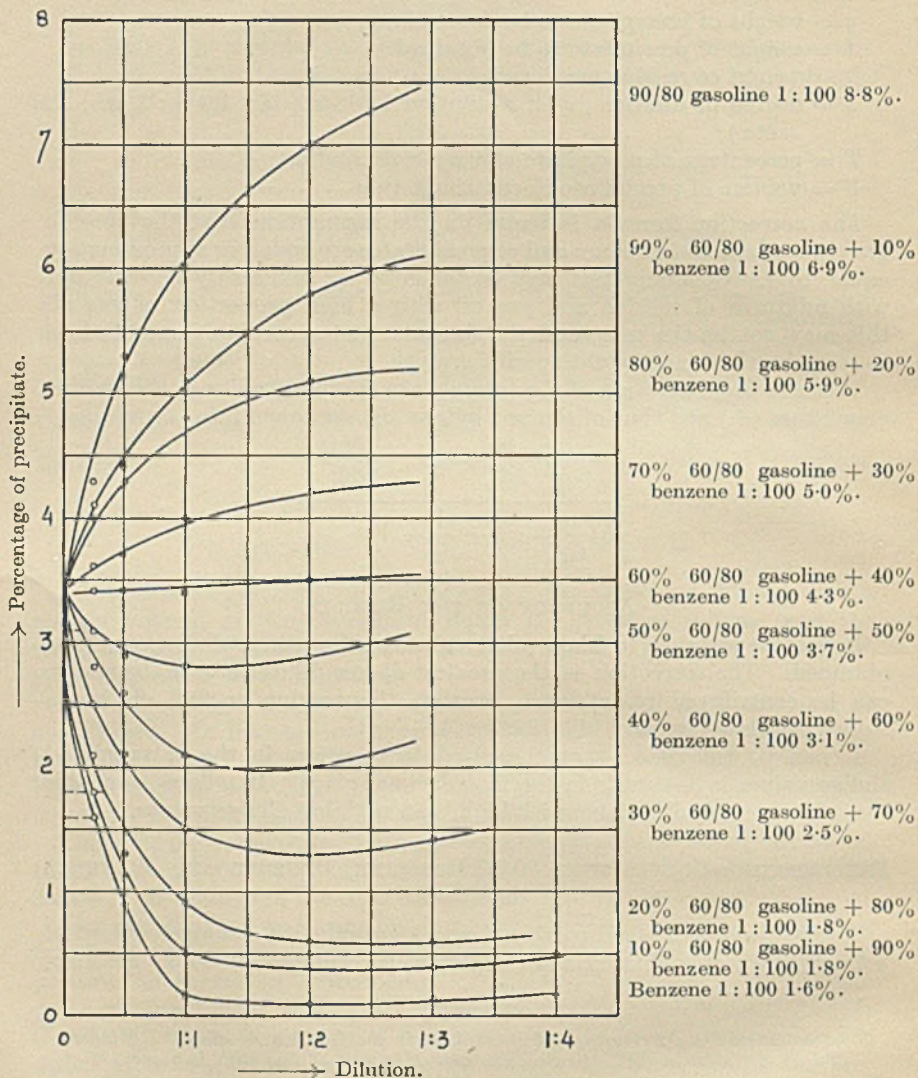


FIG. 1.

PRECIPITATION CURVES OBTAINED WITH 60/80 GASOLINE, BENZENE AND MIXTURES OF THESE PRODUCTS IN A MIXTURE OF CRACKED RESIDUE A WITH GAS OIL (40%).

benzene and mixtures of these. The resulting percentages of precipitate have been given in Table II and the precipitation curves in Fig. 1.

The precipitates had, of course, to be washed with 60/80 gasoline, as otherwise the first part of some of the curves would be similar to the dotted lines drawn in Fig. 1. Washing with the precipitant itself would be permissible only in those cases in which the content of precipitate at high degrees of dilution would be higher than the sludge content.

It is interesting to note that with the mixture 50 per cent. of 60/80 gasoline and 50 per cent. of benzene a minimum occurs in the precipitation curves. This minimum becomes more pronounced with increasing percentages of benzene in the mixture.

The effect of the benzene on the sludge is interesting. At a definite dilution there is a maximum solubility of the sludge in the medium. When more benzene is added, more precipitate is formed, reaching with infinite dilution a certain limit, just as it did with 60/80 gasoline. The obvious question now arising is whether every cracked residue, even one containing no sludge at all, behaves similarly. This is actually found to be the case. Every Dubbs residue examined by the author produces asymptotically more precipitate with increasing quantities of benzene. A minimum is, of course, absent in the case where there is no sludge. The maximum stability of a system without sludge would have to be determined by some other means.

*cyclo*HEXANE AS PRECIPITANT.

*cyclo*Hexane is a precipitant which flattens out the curves and, as we know by experience, gives no minima. The results have been collected in Table III; the percentages practically coincide with those obtained with mixtures of 70 per cent. of 60/80 gasoline and 30 per cent. of benzene and which are plotted in Fig. 1.

TABLE III.

Relation between the Percentage of Precipitate in the Residue A—Gas-Oil Mixture and the Dilution with cycloHexane.

Dilution.	Percentage of precipitate.
1 : $\frac{1}{4}$	3.6
1 : $\frac{1}{2}$	3.8
1 : 1	3.9
1 : 2	4.3
1 : 100	5.5

Table IV gives the percentages of precipitate obtained with *cyclo*hexane in different dilutions in some cracked residues. The results have been plotted in Fig. 2.

TABLE IV.

Relation between the Percentage of Precipitate in Some Residues and the Dilution with cycloHexane.

Dilution.	Residue B.	Residue A.	Residue C.	Residue D.	Residue E.	Residue F.
1 : 1	16.0%	4.0%	2.2%	0.5%	0.2%	0.2%
1 : 2	17.0	5.3	3.0	0.6	3.5	3.0
1 : 3	18.1	6.2	3.5	—	5.1	4.2
1 : 5	—	7.3	4.1	1.3	6.6	5.5
1 : 100	19.6	9.0	4.9	3.2	8.4	7.5

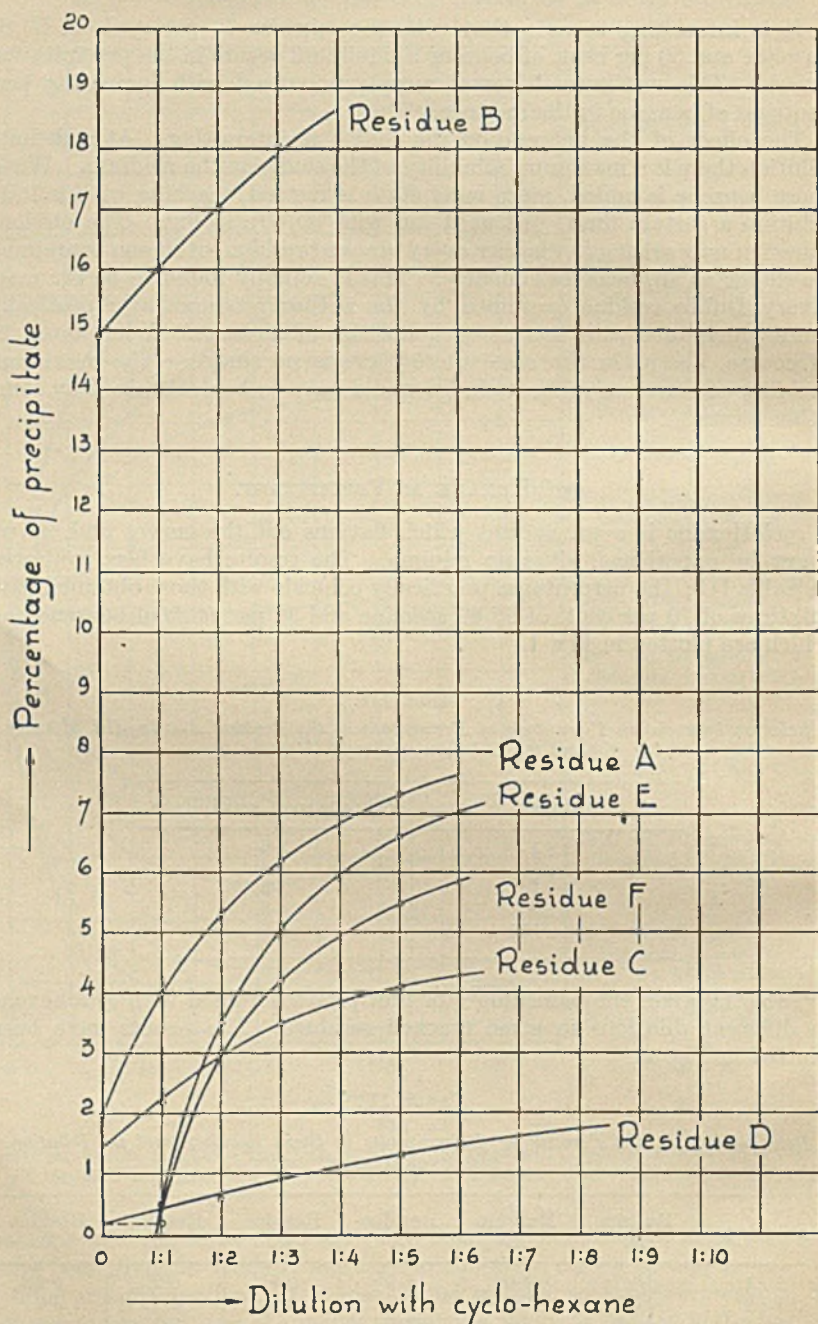


FIG. 2.

PRECIPITATION CURVES OBTAINED WITH *cyclohexane* IN A FEW CRACKED RESIDUES.

Residue *B* was a strongly cracked residue. The residues *E* and *F* were two cracked residues which could be mixed with 10–20 per cent. of P.D. bottoms without giving any difficulty in the preheaters; these are cases in which a "threshold value" was found for the formation of precipitate in cracked residue by dilution. Under the microscope, with dark-field illumination, inhomogeneities such as inorganic and carbon-like material were, indeed, noticeable, but the quantity was so small as to defy determination. It was roughly estimated to be less than 0.1 per cent. Strictly speaking, these curves of the residues *E* and *F* should not end in the *X* axis, but should run horizontally to the *Y* axis from about 0.1 per cent. (see dotted line).

The following is also worth mentioning :

In practice Residue *C* was found to be less stable towards heating than Residue *A*, although the former contains less sludge; its precipitation curve is flatter than that of *A*.

THE EXTRACTION OF A RESIDUE WITH PURE BENZENE.

The behaviour of benzene towards cracked residues and blends is of practical importance because it explains the phenomena encountered when carrying out, for instance, the Sediment-by-Extraction Test, (A.S.T.M. Designation D473–38T).

Suppose that a residue or blend (*e.g.*, the mixture of Residue *A* with gas oil) is subjected to the Sediment-by-Extraction Test.

Owing to its comparatively high viscosity, this fuel does not pass through the pores of the alundum thimble. If benzene condenses in the thimble, the composition of the fuel–benzene mixture will shift to the right along the *X* axis (see Fig. 1). If, for instance, the viscosity of the 1 : 2 mixture is such that it can pass through the pores of the alundum thimble, it will be clear that practically no precipitate is left behind.

At the bottom of the extractor, however, precipitate is deposited owing to the excess of benzene, a symptom that has frequently been noticed previously.

The quantity of sediment obtained in this test therefore depends on the pore size of the alundum thimble and the viscosity of the fuel.

SOXHLET EXTRACTION WITH SOLVENTS INCREASING IN SOLVENT POWER.

It is different when the extraction is carried out successively with solvents increasing in solvent power. This takes place in the examination of asphaltic bitumen in the Soxhlet apparatus. Hillman and Barnett have reported on this in their publication on an investigation into cracked and uncracked asphaltic bitumens.²

Hillman and Barnett separate asphaltic bitumen by means of *isopentane* into a solid and a liquid part (pentane-asphaltenes and pentane-maltenes). They next extract the pentane-asphaltenes successively with *isopentane*, hexane, carbon tetrachloride, benzene, carbon disulphide, and pyridine in a Soxhlet apparatus. The influence of the medium on, for instance, the benzene-insoluble is eliminated here by a gradual removal of the "lighter"

constituents, which, in combination with the benzene, have a maximum solvent power for the benzene-insoluble.

According to an unpublished statement of Hillman and Barnett, the percentage of carbon tetrachloride-insoluble is higher if the extraction with hexane is not immediately followed by that with carbon tetrachloride, but with mixtures of hexane and carbon tetrachloride, each time containing 10 per cent. more of the latter substance.

COMPARISON OF THE QUANTITY OF BENZENE-INSOLUBLE AFTER THE SOXHLET EXTRACTION AND AFTER HIGH DILUTION.

It has been ascertained that the quantities of benzene-insoluble in high benzene dilution and after the modified Soxhlet extraction described above are the same. One would expect this, as the influence of the medium on the benzene-insoluble is eliminated in both methods.

Table V gives the quantities of benzene-insoluble in some residues with the dilution method and after the Soxhlet extraction.

TABLE V.
Percentage of Benzene-insoluble with the Soxhlet Extraction and with the Dilution Method.

Residuc.	Percentage insoluble in pentane.	Percentage insoluble in benzene (Soxhlet extraction).	Percentage insoluble in benzene (high dilution).
A	16.2	6.5	2.3
C	8.7	2.0	0.6
D	10.0	2.9	0.4
E	15.1	4.8	0.6
H	19.2	8.7	1.8
K	20.1	10.4	2.3

TABLE VI.
Experiments with Residue A.

Percentage benzene-insoluble in accordance with the dilution method.	Percentage benzene-insoluble in accordance with the Soxhlet extraction.
(a) Dilution 1 : 100 2.3%	(d) At condensation temperature of the solvent 6.4%
Dilution 1 : 1000 2.4%	At room temperature ("cold extraction") 7.7%
(b) Dilution 1 : 100 after precipitation of the pentane insoluble <i>without</i> drying of the precipitate 2.6%	(e) Extraction in carbon dioxide atmosphere 5.9%
(c) Ditto with drying of the pentane-insoluble (18 hrs. at 110° C.) 2.9%	(f) Extraction with exclusion of light 5.7%
	(g) Extraction with 60/80 gasoline-benzene mixtures (rising by 5%) 4.9%

TABLE VII.

Shaking-out of the Pentane Precipitate with Solvents.

(a) Successively shaking-out of the pentane-precipitate with a large excess of 60/80 gasoline-benzene mixtures (rising by 5%) without drying of the precipitate	4.4%
(b) Ditto with drying of the pentane precipitate	5.5%
(c) Ditto with gasoline- CCl_4 mixtures and with benzene	5.8%

There is a remarkable difference in benzene-insoluble between the extraction and the dilution method, the results of the latter being much lower.

To account for this great difference, the author experimented further with Residue *A*.

The idea was to vary several factors (such as drying of the pentane precipitate, influence of light and oxygen, etc.) which might influence the results of the dilution and the extraction method. The results of these experiments have been collected in Tables VI and VII.

Experiments with the Dilution Method.

(a) In the first place it was ascertained whether 1 : 100 was a sufficient dilution. To this end the percentage of precipitate was determined at a dilution of 1 : 1000. The increase of 0.1 per cent. (2.3–2.4 per cent., *a*, Table VI) is practically negligible. In point of fact we had already found that, in a 1 : 100 dilution, every cracked residue is on the asymptotic part of the precipitation curve.

(b) It had occurred to us that the previous precipitation and drying of the pentane-insoluble might cause such changes in the precipitate as to render part of it insoluble in benzene. The drying was first eliminated by at once taking up the filtered pentane precipitate in excess benzene. Although this gave an increase of 0.3 per cent., the value for the benzene-insoluble was still far below 6.4 per cent.

(c) The influence of drying was ascertained by drying the pentane precipitate for 18 hours at 110° C. By this manipulation the value was slightly increased to 2.9 per cent.

Experiments with the Extraction Method.

(d) It might be that normal extraction at the rather high condensation temperature of the solvents could effect polymerization or the like of the residue constituents, causing these to become less and less soluble.

If so, extraction at room temperature should give lower results. This "cold extraction" was carried out as follows (see Fig. 3) :—

The separating funnel *A* containing the extraction liquid was connected by means of a glass tube with drain-cock *F*, with a wide glass filter *B*, which contained a Jena Glass filter *G* 3 (*C*) with the precipitate. *A* was heated by an electric heating ring *D*. The solvent vapours consequently condensed in the condenser *E* and the liquid regularly dripped into *C*. Extract plus solvent were conducted back to *A* through the glass tube.

After the cold extraction the percentage benzene-insoluble was 7.7 per cent., hence *higher* than after the Soxhlet extraction. Also in cold ex-

tractions with the other residues the percentages of benzene-insoluble were either higher than or equal to those after the Soxhlet extraction. The high temperature was consequently not responsible for the great difference between the dilution method and the extraction method.

(e) The possible influence of oxygen on the precipitate (oxidation polymerization) was next considered, as during the long time required by the Soxhlet extraction the precipitate has ample opportunity of being oxidized. An extraction carried out in a current of carbon dioxide actually gave a lower result : 5.9 per cent., but this was still much higher than that of the dilution method.

(f) The percentage of benzene-insoluble was not radically altered

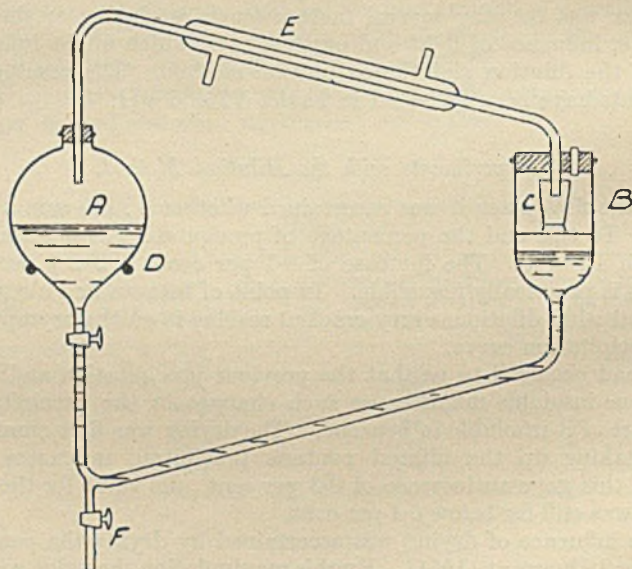


FIG. 3.

EXTRACTION APPARATUS.

by the influence of light. An extraction carried out in the dark gave a result of 5.7 per cent.

(g) Carbon tetrachloride is known to react with a number of substances (bituminous substances among them) under the influence of light, splitting off HCl .^{3, 4}

A well-known example of this is the decomposition of CCl_4 in the presence of iron. When a little aniline is boiled with CCl_4 under a reflux condenser in diffused daylight, hydrochloric acid can be distinctly detected after half an hour.

Dubbs residue and "asphaltenes" also gave a positive chlorine reaction after being boiled with CCl_4 for half an hour under a reflux condenser. As liberated chlorine and chlorine compounds cause such changes in residues as to render them less soluble (coagulation, polymerization), it is natural

to assume that the great difference between extraction and dilution method might be due to the use of CCl_4 . An extraction was therefore made without CCl_4 .

If CCl_4 is omitted, the extraction with 60/80 gasoline should be followed by extraction with mixtures of 60/80 gasoline and increasing quantities of benzene (rising by 5 per cent.), and finally with pure benzene. By this procedure the author obtained 4.9 per cent. of benzene-insoluble—i.e., 1.5 per cent. lower than after extraction with CCl_4 .

This effect is, however, incapable of accounting for the difference from the dilution method, which is still large.

The only explanation left was to assume that the effect of step-by-step removal of the protection round the pentane precipitate so as to eliminate the influence of the soluble on the insoluble part differs fundamentally from the application of a high dilution to attain the same purpose.

This was further demonstrated by the following two experiments :

(1) *Shaking out of the pentane precipitate with solvents of an increasing solvent power.*

(2) *Mixing experiments.*

(1) *Shaking out the Pentane Precipitate with Solvents.*

(a) *With Gasoline-Benzene Mixtures.*—This manipulation may be considered as intermediate between the dilution and the extraction method.

The residue was weighed into the centrifuge tubes and diluted with $100 \times$ its volume of pentane. The pentane precipitate centrifuged off was thoroughly washed a few times with pentane until the wash liquid was almost colourless. The precipitate was subsequently mixed with 60/80 gasoline and the above manipulations were repeated. The gasoline was replaced by a mixture of 95 per cent. 60/80 gasoline and 5 per cent. benzene, and so on, ending with pure benzene. The benzene-insoluble was then rinsed out with CS_2 into a weighed gum dish; after evaporation of the CS_2 the precipitate was dried and weighed. The percentage insoluble was 4.4 per cent. (a, Table VII).

This shows that the shaking method gives a final result very close to that obtained in the extraction method with gasoline-benzene mixtures.

(b) A previous drying of the pentane asphalthenes immediately caused this figure to rise to 5.5 per cent. (b, Table VII).

(c) The influence of CCl_4 on the shaking method was also ascertained. The residue was shaken first with 60/80 gasoline and subsequently with mixtures of 60/80 gasoline and CCl_4 . As the mixture with 80 per cent. of CCl_4 did not separate in the centrifuge owing to the slight difference in specific gravity between precipitate and liquid, the precipitate obtained after centrifuging with 70 per cent. of CCl_4 and 30 per cent. of 60/80 gasoline and washing out was mixed with pure CCl_4 . This precipitate was suspended in the CCl_4 by shaking, part of it dissolved, and the insoluble part then had a structure which allowed it to be filtered off in a Jena-glass filter G 4. The residue on the filter was thoroughly washed with CCl_4 , then rinsed quantitatively into the centrifuging tube and shaken out with benzene. In spite of the fact that a few stages had been skipped in the shaking

process, and that a slight loss might have occurred in rinsing the CCl_4 precipitate into the centrifuge tube, the percentages of benzene-insoluble now amounted to 5.8 per cent. (c, Table VII).

(2) *Mixing Experiments.*

Residue *A* was mixed in different concentrations with a straight-run residue of an "aromatic" type and with pyridine. The percentage benzene-insoluble in the mixtures was determined with the dilution and with the extraction method. The results are collected in Table VIII and in Fig. 4.

TABLE VIII.

Percentages of Benzene-insoluble in Mixtures of Residue A with a Straight-run Residue and with Pyridine according to the Extraction and the Dilution Methods.

Mixture.	Extraction.	Dilution.	Mixture.	Extraction.	Dilution.
100% residue <i>A</i>	0.4%	2.3%	100% residue <i>A</i>	0.4%	2.3%
80% res. <i>A</i> + 20% str.-run res.	6.0	1.0	80% res. <i>A</i> + 20% pyridine	5.4	1.3
60% " + 40% " "	5.2	0.4	80% " + 40% " "	3.0	0.89
40% " + 60% " "	—	0.3	40% " + 60% " "	2.6	0.59
30% " + 70% " "	3.8	—	30% " + 70% " "	—	—
20% " + 80% " "	—	0.1	20% " + 80% " "	1.3	0.23
100% straight-run residue	2.8	0.09	100% pyridine	0	0

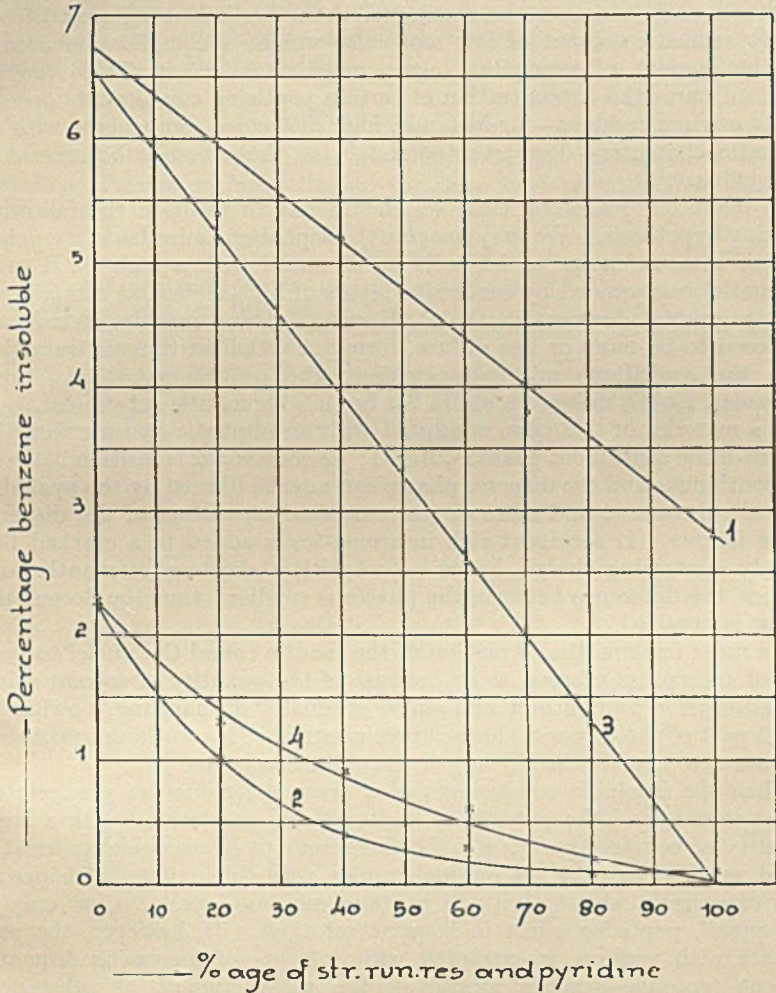
When plotted, the quantities of benzene-insoluble as a function of the mixing ratio give a straight line in the extraction method (Fig. 4, the straight lines 1 and 3); in the dilution method *curves* are obtained (Fig. 4, curves 2 and 4). In the latter method the percentage insoluble is invariably lower than the theoretical quantity. This discrepancy is more pronounced with the straight-run residue than with the pyridine mixture.

Theoretical Considerations.

In the previous publication ¹ it was stated that the compounds of straight-run and cracked residues insoluble in aromatic-free gasoline, the so-called "asphaltenes," form a large series of products with varying solubility in organic media. According to the publication of Pfeiffer and van Doormaal ⁵ the asphaltenes of bitumens dissolve or peptize better in non-polar organic liquids of a hydro-aromatic or aromatic nature than in aliphatic ones, and especially the high-molecular aromatics favour the peptization of the asphaltenes.

It will be clear that the different curves represented in Fig. 1 obtained by diluting the mixture of cracked residue *A* and a gas-oil with increasing quantities of several gasoline-benzene mixtures illustrate the relation between the aromatic content of the solvent and its solvent power on the "asphaltenes" of this residue. It appears that the solvent power of cyclohexane is about the same as that of a mixture of 70 per cent. of gasoline and 30 per cent. of benzene. As the amount of insoluble substances in these liquids is still higher than the sludge present in the fuel, we must assume that the other components of this fuel have a still better dissolving power upon its asphaltene; it could be compared with that of a mixture of, say, 58 per cent. of gasoline and 42 per cent. of benzene. This mixture

may therefore be regarded as the "neutral washing liquid" for the sludge, as was described in the previous publication,¹ p. 189.



- 1 Extraction of residue A-str. run. res. mixtures.
- 2 Dilution " " " " " "
- 3 Extraction of residue A-pyridine " " " "
- 4 Dilution " " " " " "

FIG. 4.

THE PERCENTAGES OF BENZENE INSOLUBLE IN MIXTURES OF RESIDUE A WITH A STRAIGHT-RUN RESIDUE AND WITH PYRIDINE ACCORDING TO THE EXTRACTION AND THE DILUTION METHOD.

The increasing amounts of precipitate formed when employing increasing amounts of these solvents with less dissolving power than the maltenes

must be explained by the decreasing influence of the maltenes in the higher dilutions. The same reasoning can be applied to explain the decreasing amount of the sludge with small quantities of better peptizing solvents (mixtures containing more benzene). With further increasing quantities of highly aromatic solvent of low molecular weight a complication occurs, for the amount of precipitate again increases. It is probable that in these mixtures the concentration of certain peptizing constituents present in the original residues—for instance, high molecular components with an aromatic character—decreases too much, so that these substances lose their efficiency.

To make our reasoning clear we should like to mention the following working hypothesis. We may imagine the asphaltene micellae in a cracked residue without sludge to be built up of nuclei with a high C:H ratio (aromatic), surrounded by successive layers of high-molecular components with a gradual decrease in the C:H ratio. These mantles, which are supposed to be more or less diffuse, form a kind of continuous transition from the asphaltene micella nucleus to the continuous phase. Low-molecular, mobile molecules would not be suitable mantle substances.

If a material of this type is diluted with an aliphatic hydrocarbon the nature of the continuous phase is altered; no continuous transition between the continuous and the disperse phases can now be formed by the available mantle substances, and more or less complete separation of the disperse phase follows. If a solvent rich in aromatics is added to a cracked fuel already containing sludge, hence a fuel with a shortage of mantle substances, the difference between the phases is smaller; thus the flocculated sludge is peptized.

We must imagine the formation of the mantle round the nuclei to be a sort of adsorption process, as an increase of the quantity of solvent shifts the adsorption equilibrium and cause gradual "dismantling," owing to which part of the disperse phase is precipitated. This working hypothesis may serve to explain the asymptotic precipitation curve.

When the insoluble components of a cracked residue are precipitated with pentane, and the precipitate is filtered off and treated with a large quantity of benzene, practically the same amount of insoluble material is found as when treating the original residue with this amount of benzene. This experiment shows that the pentane maltenes show no, or only a very small, peptizing effect in benzene solutions. If, however, the precipitate with pentane is extracted with solvents of increasing aromatic content (gasoline-benzene mixtures), the total amount of substances insoluble in benzene increases, showing that that part of the asphaltenes which is soluble in these mixtures is necessary to peptize part of the material in itself insoluble in benzene; these fractions of the asphaltenes must be regarded as the mantle substances of the cracked nuclei in the solution in benzene. It will now be clear that from the amount of benzene insoluble after the extraction in our mixing experiments (Table VIII) a mathematical relationship could be found between the dilution and the amount of benzene-insoluble material, because all the nuclei and their mantles were precipitated with pentane, and after that the mantles were quantitatively removed. In the dilution experiments, however, these protective bodies remain present, but their influence cannot be expressed in a simple

mathematical equation with the mixing proportion, as the adsorption equilibrium now also plays a part. For actual practice the most valuable criteria are the amount of sludge present in a fuel without dilution (extrapolated sludge value) and the changes of this quantity upon blending or heating; the experiments described, however, displayed the colloid chemical nature of Dubbs residue.

ACKNOWLEDGMENT.

The author wishes to express his thanks to the Management of the Bataafsche Petroleum Maatschappij for their kind permission to publish the foregoing.

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THE CATALYTIC EFFECT OF METALS ON THE AGEING OF LUBRICATING OIL FOR DIESEL ENGINES.*

By H. L. MATTHIJSEN †

DURING the examination at regular intervals of the used lubricating oils from the Maybach engines of the triple car-Diesel trains belonging to the Netherlands Railways, the question was raised as to whether the small metal splinters present in the used oil and also the cylinder-wall, the piston-rings, etc., might be able to increase or reduce the content of oxidation- or ageing-products.

Many references to the effect of metals, etc., on ageing are to be found in the literature (5, 8, 16, 21, 26, 31, 32, 33, 35, 36, 37, 42), but on the whole they are so vague that we have found it necessary to test this point ourselves.

As it was not possible to carry out such work in the engines of the diesel trains, the experiments were made in a laboratory oxidation instrument designed by Mr. A. Mollinger, Mech. Eng., a lecturer at the Technical High School at Delft, Mechanics Section.

We have learned from an elaborate study of the relevant literature of recent years on the ageing of lubricating oil (2, 3, 4, 5, 6, 7, 8, 10, 14, 21, 22, 24, 27, 28, 29, 32, 38, 39, 41), that twenty-three different laboratory oxidation methods have been worked out.

The B.A.M. Test and the Indiana oxidation test were preferred some time ago in the English-speaking countries, but during the past year the simple dish method has become popular and found favour with the Bureau of Standards. The Sligh test, which the A.S.T.M. at first recommended,²⁹ has recently declined in favour. France, Italy and Germany, moreover, each have methods of their own.^{6, 22, 4}

Although a good many articles make mention of satisfactory correlation between the engine tests and some or other oxidation test, considerable uncertainty still prevails, and many still hold the view that oxidation tests can never give conclusive evidence as to the quality of a lubricant in practice (cf.¹⁷).

Our principal object being to determine the catalytic effect of metals on ageing, and not the quality of the lubricating oil in practice, we are publishing these results in the hope that they may contribute something towards a better understanding of the decisive factors governing the ageing of lubricating oils. Figs. 1 and 2 represent the oxidation instrument, designed by Mollinger.

100 grams of the oil under test are put into a sheet-iron tank, after which

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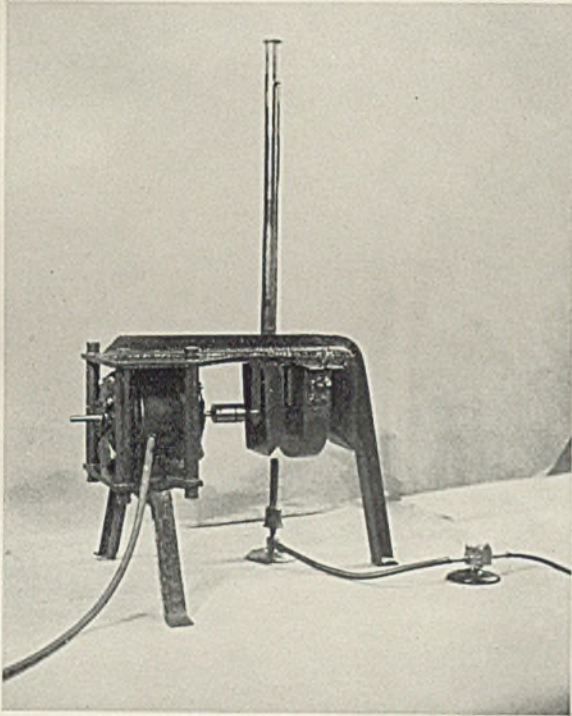


FIG. 1.
OXIDATION INSTRUMENT DESIGNED BY MOLLINGER.

this is fitted to the upper part of the apparatus. Then the saw, driven by an electric motor at 2800 r.p.m., is made to rotate. As a result, the oil is flung in minute particles towards the chimney and flows back along the sides into the tank. At the same time the gas-burner under the tank is lit. As soon as the dispersed oil has attained the temperature of the test, this temperature is kept constant for 85 minutes, which is quite feasible up to 230° C. with a fluctuation of $\pm 1^\circ$ C.

As is evident from Fig. 2, a certain amount of air, passing between the two loosely-fitted rings rotating with the axle and the sides of the tank, will reach the oil and promote oxidation. The oil fumes escape through the chimney, the upper part of which is provided with four slanting partitions. Oil fumes also escape through the small apertures between the rotating rings and the sides of the tank. The thermometer is placed almost immediately above the gas-burner, projecting 5.8 cm. into the tank. The velocity with which the oil moves at the bottom of the tank makes superheating impossible.

At the end of the ageing test a short interval is allowed for the oil to cool, if necessary, after which the tank with the oil is weighed to the nearest 0.5 gm. in order to determine the loss by evaporation. The aged oil is then further examined.

We have now aged seventy-eight different lubricating oils for diesel engines at 230° C. in the oxidizer described and illustrated here.

We preferred this high ageing temperature because we consider this to be right for diesel engines. This opinion is corroborated by Mr. Fairlie's citation³² of 232–371° C. for piston temperatures, all according to the load.

As it has not yet been possible to put several of the above-mentioned seventy-eight lubricating oils through engine tests, we have to be very careful in interpreting the results of these ageing tests, and we must not attach a definite value to them as regards a quality test for service.

Although many indications have been provided, we do not yet wish to call them proofs.

Thus one of the two kinds of lubricating oil used in our diesel engines thickens more than the other; this agrees with the oxidation test. It is furthermore evident from the results of the oxidation tests with the seventy-eight lubricating oils that the pure Pennsylvania oils age least of all and are in this test superior to the naphthene base oils. This fact is borne out by several statements in the relevant literature^(21, 25, 27, 32, 39, 40). It was also found that many solvent-treated oils age considerably. This finds corroboration in some publications and refutation in others^(11, 32, 34, 40).

Besides the ageing test in our instrument, the Indiana oxidation test was also carried out with forty-one different oils in a co-operating laboratory.

Appraised and classified in the same way, both oxidation tests of twenty-seven kinds of oil correlate fairly well, whereas with the remaining fourteen oils the comparison turns out rather badly. This unfavourable result is not surprising, since the method of ageing is quite different.

In the literature the oxidation tests often tally well with the corresponding engine tests^(2, 5, 9, 13, 15, 30, 35). On the other hand, however, several oxidation tests are mentioned which do not agree^(4, 17, 18).

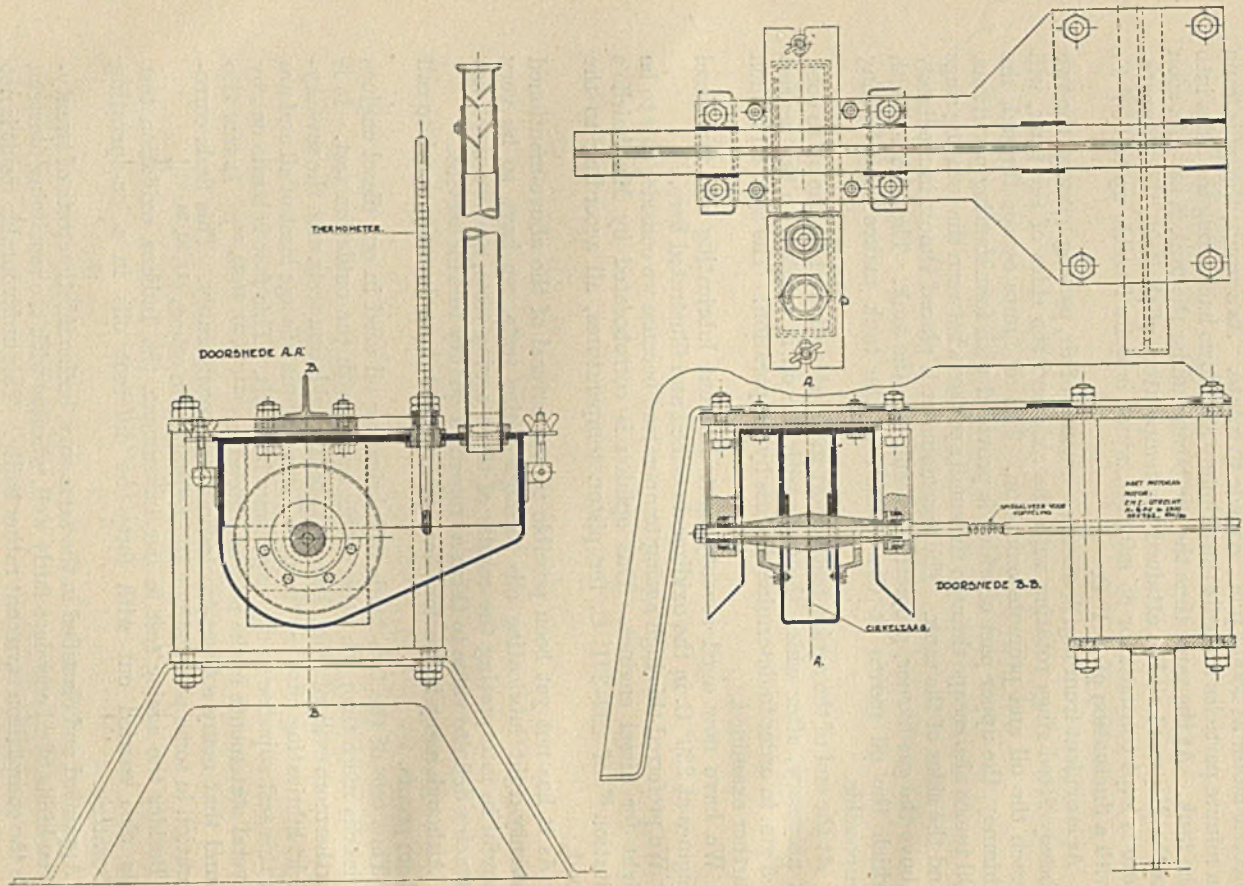


FIG. 2.

PLAN OF OXIDATION INSTRUMENT DESIGNED BY MOLLINGER.

The lubricating oil aged in the instrument is tested for :

- (1) sludge (soluble in benzene) ;
- (2) viscosity at 100° C. in centipoises ;
- (3) acid number ;
- (4) saponification number ;
- (5) Conradson residue.

SHORT DESCRIPTION OF THE METHODS OF TEST.

(1) The sludge content is determined by adding 5 gm. of oil to 200 cu.cm. of petroleum ether (boiling range 40–60° C.). The liquid is kept for about 24 hours in a dark place, after which, by repeated centrifuging with petroleum ether, the sludge is weighed in the tube after drying for 1 hour at 105° C.

(2) The viscosity at 100° C. is determined by the Vogel–Ossag viscometer with an excess pressure of 60 cm. of water.

(3) For the acid number 100 cu. cm. of neutralized 96 per cent. alcohol is added to the weighed quantity of oil (about 4 gm.) in an Erlenmeyer flask. After boiling for a short time and cooling for 10 minutes, the solution is filtered. The filtrate is titrated with $\frac{1}{10}N$ -KOH with phenolphthalein as indicator.

(4) For the saponification number 25 cu.cm. of alcoholic KOH are added to the weighed quantity of oil (about 2 gm.) in an Erlenmeyer flask. The mixture is then boiled for an hour, a long glass tube serving for cooler. It is then titrated with 0.5*N*-hydrochloric acid and phenolphthalein as indicator ; after the turning-point to colourless is reached, it is boiled for a further 5 minutes and again titrated after more drops of the indicator have been added. A blank test is made at the same time.

The saponification number is :

$$\frac{\text{cu. cm. 0.5N-HCl for blank test} - \text{cu. cm. 0.5N-HCl for determination}}{\text{weighed quantity of oil}} \times 56.16.$$

(5) The Conradson residue is determined by the prescribed method with the standard apparatus for the test.

In Table 1 are given the different metals with which the lubricating oil comes into contact in the Maybach diesel engines and their chemical composition.

Minute turnings of these twelve different metals were cut with a diamond-cutter. The catalytic effect of each of these metals on the ageing is determined by adding in each case 0.3 per cent. of the turnings, in order to age the lubricating oil in the above-mentioned oxidation instrument at 230° C. for 85 minutes.

Although not all the metals in the engine will come into contact with the lubricating oil at 230° C., the temperature is nevertheless kept the same in all the tests, in order at any rate to be able to make a comparison at 230° C.

It goes without saying that after each test the tank, the saw, etc., are thoroughly cleaned, so that not a single metal particle from the preceding test shall be left behind. All the tests were duplicated. Before analysing

TABLE I.

	Cylinder cast iron A.	Cylinder cast iron B.	Cast iron of piston-rings.	Copper containing silumin of the pistons.	Copper containing silumin of the crank-case.	Chromium steel of roller main bearing.	Chromium nickel tungsten steel of roller connecting rod bearing.	Chromium nickel tungsten steel of race connecting rod bearing.	Aluminium manganese bronze of cage connecting rod bearing.	Chromium nickel steel of pumping wheel.	Red copper of tube oil filter.	Brass of wire cloth from oil filter.
Phosphorus, % . . .	0.100	0.112	0.580	—	—	—	—	—	—	—	—	—
Sulphur, %	0.136	0.115	0.086	—	—	—	—	—	—	—	—	—
Manganese, %	0.84	0.84	0.63	0.46	0.16	0.30	0.41	0.36	3.3	0.42	—	—
Silicon, %	2.34	2.36	2.60	13.3	13.4	0.19	0.26	0.28	—	0.24	—	—
Total carbon, %	3.30	3.26	3.40	—	—	1.0	0.70	0.62	—	0.34	—	—
Graphite, %	2.62	2.56	2.91	—	—	—	—	—	—	—	—	—
Combined carbon, %	0.68	0.70	0.49	—	—	—	—	—	—	—	—	—
Chromium, %	—	—	—	—	—	1.4	1.14	1.3	—	0.66	—	—
Nickel, %	—	—	—	—	—	nil	4.35	4.1	—	3.28	—	—
Tungsten, %	—	—	—	—	—	traces	0.64	0.41	—	nil	—	—
Copper, %	—	—	—	1.40	1.0	—	—	—	88.2	—	rest	64.6
Zinc, %	—	—	—	—	—	—	—	—	traces	—	nil	35.2
Tin, %	—	—	—	—	—	—	—	—	traces	—	nil	nil
Lead, %	—	—	—	nil	nil	—	—	—	traces	—	nil	nil
Magnesium, %	—	—	—	0.52	0.11	—	—	—	—	—	—	—
Iron, %	—	—	—	3.50	0.96	—	—	—	0.4	—	nil	nil
Aluminium, %	—	—	—	rest	rest	—	—	—	8.2	—	nil	—
Arsenic, %	—	—	—	—	—	—	—	—	—	—	traces	—

A striking effect is the pronounced counteracting effect on the ageing of the copper of the tube oil-filter, and especially of the aluminium-manganese-bronze of the cage connecting rod-bearing.

(6) On the whole there is some connection between the alterations in viscosity and those of the sludge content and the Conradson residue. The saponification and acid numbers do not form a steadily decreasing series and have little connection with each other. The loss by evaporation is scarcely affected by the addition of metals. It is only with copper and aluminium manganese-bronze, which counteract the ageing considerably, that there is less loss by evaporation.

The question raised at the beginning of this paper, viz., whether the metal splinters present in the used lubricating oil and also the cylinder wall, piston-ring, piston, etc., will change the content of oxidation products, may be answered in the affirmative.

As a matter of course, it is mainly the iron metals which will come into contact with the lubricating oil in the engine. The catalytic effect of these metals is doubtless positive; consequently the lubricating oil will age more and the oxidation products will increase.

As has been previously mentioned, a striking fact is the strong inhibiting effect copper has upon the ageing of the lubricating oil at 230° C. It is, however, known that copper is deleterious⁴³; e.g., this is the case with transformer and turbine oils.

The temperature to which transformer oil especially is exposed, being much lower than 230° C., we have extended the test with copper in order to determine the catalytic effect of an addition of 0.3 per cent. of minute turnings on the ageing at rising temperature, viz. at 100° C., 140° C., 180° C., 183° C., 187° C., 193° C., 205° C., 218° C., 224° C. and 230° C.

In order to ascertain whether the catalytic effect of copper is the same on the ageing of different kinds of lubricating oils, we carried out the same experiment with a naphthene base oil. The above-mentioned temperatures have been purposely chosen, because the series of tests with the paraffin-base oil required this, as will be evident below.

The specifications of the two lubricating oils under test are tabulated below.

Determinations.	Lubricating oil A on paraffin base.	Lubricating oil B on naphthene base.
Specific gravity at 15° C.	0.886	0.912
Flash point (open cup)	243° C.	223° C.
Fire point (open cup)	292° C.	274° C.
Viscosity at 20° C.	654.9 c.p. = 97.7° E.	655.5 c.p. = 95° E.
" 50° C.	91.1 " = 15.2° E.	79.7 " = 11.8° E.
" 100° C.	14.5 " = 2.61° E.	10.7 " = 2.08° E.
Hard asphalt	nil	nil
Soft asphalt + paraffin wax	"	"
Foreign matter	"	"
Loss of weight on heating to 150° C.	0.03%	0.04%
Pour point (Neth. Railways method)	-17° C.	-19° C.
Acid number	0.17	0.26
Conradson residue	0.89%	0.41%
Sulphur	0.20%	0.87%
Anilino point	117.8° C.	92.0° C.

The tests were all carried out in duplicate, the first series, from 100° C. to 230° C., being finished off first and then the second. Between the two series the instrument was thoroughly cleaned.

In each case the specific gravity at 15° C., the sludge content (soluble in benzene), the viscosity at 100° C. in c.p., the Conradson residue, the acid number and the saponification number of the aged lubricating oil were determined.

The methods of test have already been described in this paper. The specific gravity is determined with a pyknometer.

Of the whole process of each of the six determinations a graph has been made showing the changes owing to the ageing at rising temperature of the lubricating oil without and with the addition of 0.3 per cent. of minute copper turnings.

The research results with lubricating oil *A* are represented in Figs. 3, 4, 5, 6, 7 and 8.

From these graphs it is evident first of all that the catalytic effect of the added copper on the ageing of the lubricating oil at lower temperatures is quite different from that at higher temperatures. Apart from the sludge which begins to form above 218° C. in the lubricating oil (without copper) in the case of the five remaining determinations, the catalytic effect of copper at 180° C. changes from clearly but weakly positive to strongly negative. This result removes the contradiction noted with copper (see Table II). The above-mentioned results accord more or less with the statements of A. Maillard⁸ and M. Moutte, Dixmier and Lion.¹⁶

It is evident from the shape of the graphs that Fig. 4 (sludge) and Fig. 5 (viscosity) are closely connected, which is also the case with Fig. 3 (specific gravity), Fig. 6 (Conradson residue), Fig. 7 (acid number) and Fig. 8 (saponification number).

The two first-mentioned graphs show that above 218° C. the viscosity of the oil without copper is raised considerably, owing to a rapidly increasing sludge content.

As the copper represses the formation of sludge very considerably, the negative catalytic effect on the viscosity is most clearly shown at 230° C.

The course of the viscosity is, moreover, affected by the loss by evaporation, which increases gradually up to 5 per cent. as far as 205° C. for the oil without copper and then jumps suddenly at 218° C., 224° C. and 230° C. to 15.5 per cent., 16 per cent. and 21.5 per cent.

Up to 205° C. copper does not show any effect, whereas at 218° C., 224° C. and 230° C. the loss by evaporation falls to 9.5 per cent., 14 per cent. and 18.5 per cent.

As regards the other four determinations, the negative catalytic effect of copper increases from 180° C. to 218° C. rather quickly on the whole. Above 218° C. the anti-oxidizing effect of copper rapidly decreases.

The connection between the acid number and the saponification number, moreover, stands out clearly in Figs. 7 and 8.

The sharp fall in the acid number between 180° C. and 183° C. is worthy of note. The specific gravity likewise substantially decreases (Fig. 3).

It may finally be inferred from the fact of the similarity between the shapes of the graphs of the specific gravity and the Conradson residue (respectively Fig. 3 and Fig. 4) that a connection exists between the two

determinations. This connection emerges equally clearly from the oil with or without the addition of copper.

Below we give the results of the tests obtained on lubricating oil B, represented in Figs. 9, 10, 11, 12, 13 and 14.

In absolute contrast to lubricating oil A, copper in lubricating oil B has only a slight catalytic effect on the ageing. With all the graphs the dotted line runs closely along the drawn line, or winds round it several times. Only in Fig. 10 (sludge) does a turning-point become clearly visible at approximately 197° C. However, the change here is from negative to positive, although the catalytic effect of copper is slight, below as well as above 197° C.

With this lubricating oil there is no similarity between the viscosity and sludge curves (Fig. 11 and Fig. 10).

The pronounced increase in the sludge content, beginning at 205° C. does not entail considerably increased viscosity, although the loss by evaporation at the higher temperatures increases a good deal more than with lubricating oil A.

Accordingly Figs. 13 and 14 show much less similarity than Figs. 7 and 8 with lubricating oil A.

The only analogy to lubricating oil A is the similar shape of the curves of the specific gravity (Fig. 9) and the Conradson residue (Fig. 12).

Here again, then, is found some connection between the course of these two determinations.

Needless to say, copper also only very slightly affects the loss by evaporation.

The two above-mentioned tests prove convincingly that the catalytic effect of copper may be practically nil, but also weakly positive as well as very strongly negative, all according to the kind of lubricating oil used and to the ageing temperature. The fact that the catalytic effect on the ageing depends very much on the kind of lubricating oil used has been previously noticed by E. W. J. Mardles.²¹

Finally, let us try to realize what reactions come into play while the two lubricating oils A and B are ageing in the oxidation apparatus designed by A. Mollinger.

It is stated in the literature (1, 4, 6, 9, 10, 11, 12, 16, 19, 20, 21, 23, 24, 25, 31) that the following reactions may occur during the ageing of lubricating oil :

(1) Oxidation involving primarily the formation of acids, and secondarily what are known as petroleum resins, etc., proceeding by condensation from these acids.

At higher temperatures asphaltic matter originates, formed entirely or in part at the expense of the acids and the petroleum resins. This asphaltic matter is completely or partly soluble in the lubricating oil.

(2) Condensation or polymerization. Although there is a difference of opinion as to which of these two reactions takes place, we shall speak about condensation.

Explanation of Figs. 3-8.

- = Lubricating oil A without addition.
 - - - - - = Lubricating oil A with 0.3% of very fine copper turnings.
 ○ = Lubricating oil A original before ageing.

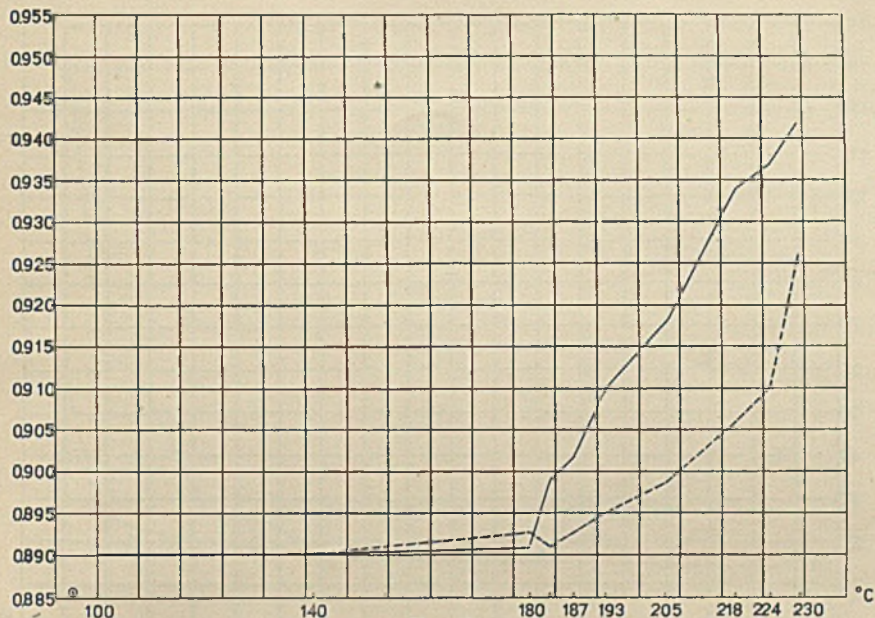


FIG. 3.

CHANGE IN SPECIFIC GRAVITY AFTER AGEING AT DIFFERENT TEMPERATURES.

% Sludge

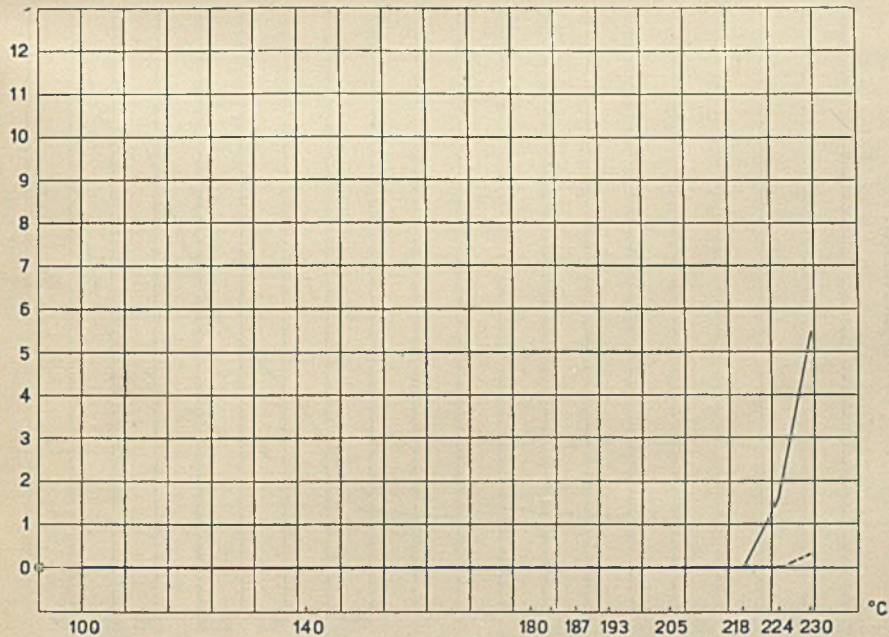


FIG. 4.

FORMATION OF SLUDGE (SOLUBLE IN BENZENE) AFTER AGEING AT DIFFERENT TEMPERATURES.

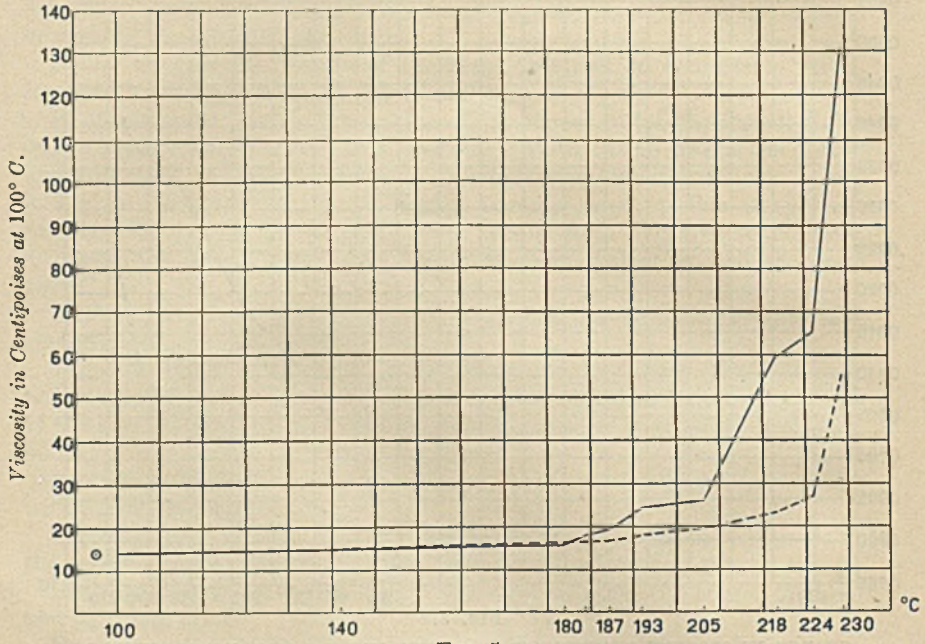


FIG. 5.

CHANGE IN VISCOSITY AT 100° C. AFTER AGEING AT DIFFERENT TEMPERATURES.

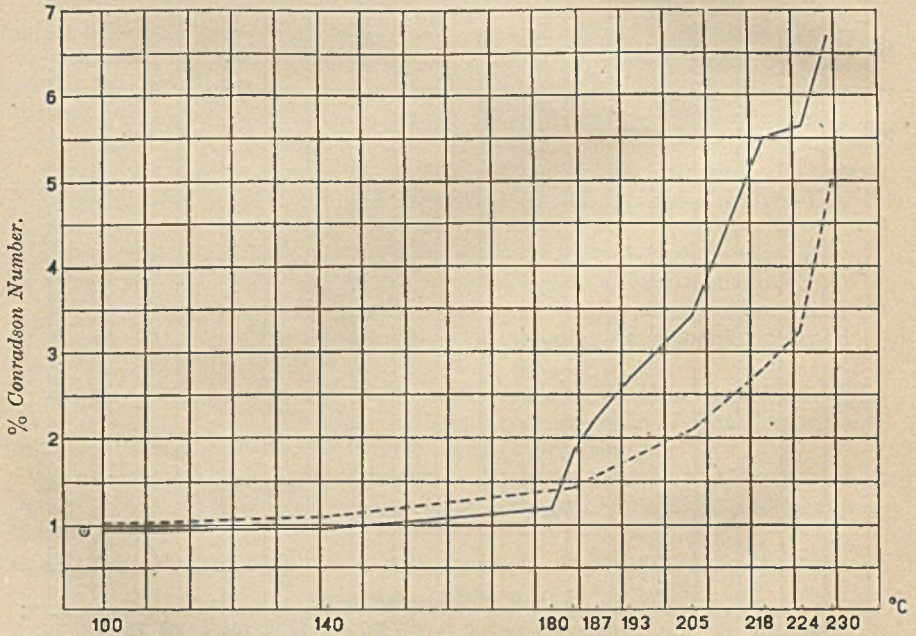


FIG. 6.

CHANGE IN CONRADSON CARBON RESIDUE AFTER AGEING AT DIFFERENT TEMPERATURES.

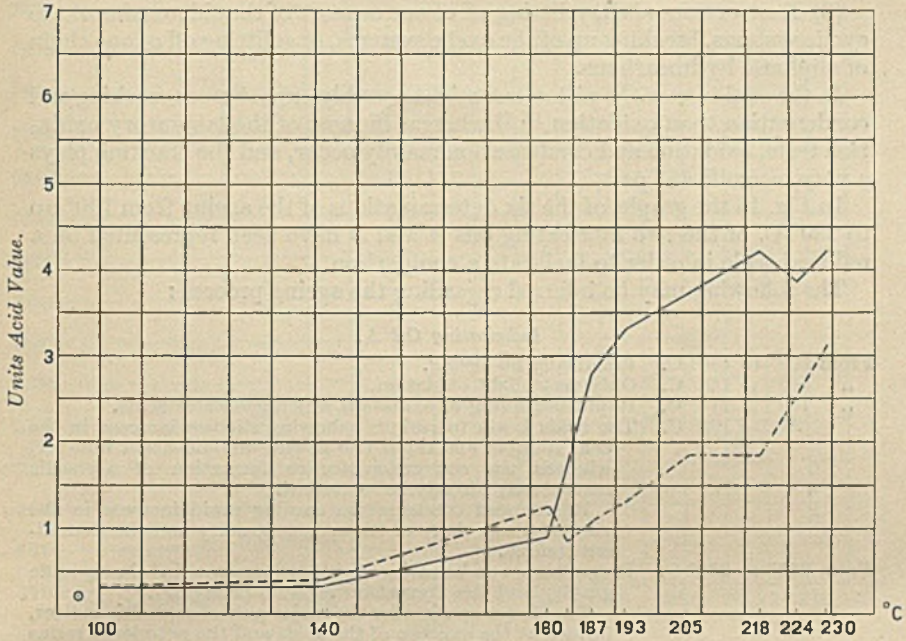


FIG. 7.

CHANGE IN ACID VALUE AFTER AGEING AT DIFFERENT TEMPERATURES.

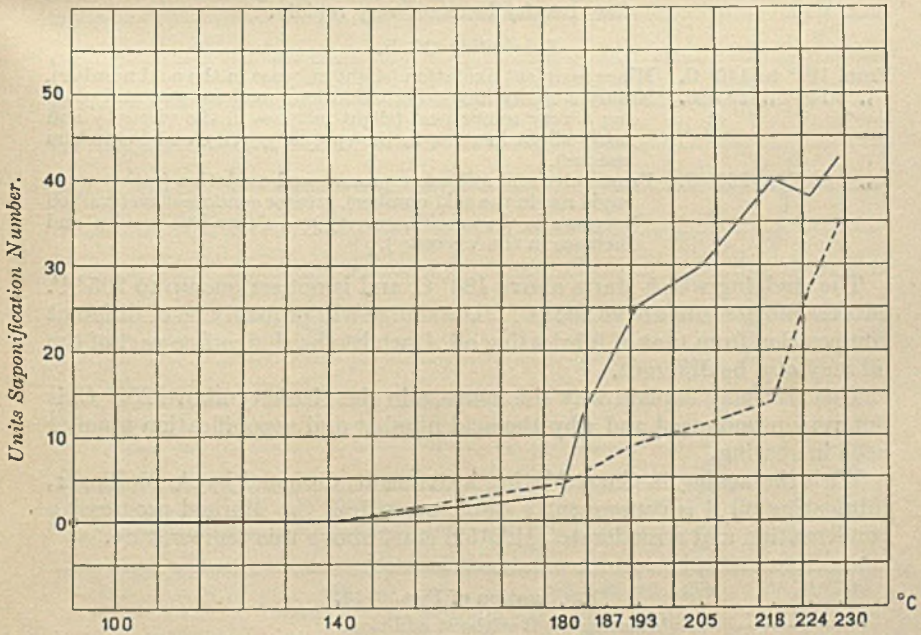


FIG. 8.

CHANGE IN SAPONIFICATION NUMBER AFTER AGEING AT DIFFERENT TEMPERATURES.

(3) Destruction—*i.e.*, splitting off of one or more of the side-chains of the cyclic systems, breaking up of the cyclic systems, or splitting off of one chain of aliphatic hydrocarbons.

In the engine near the piston one might possibly expect more cracking and condensation than oxidation,^{19, 23} whereas in most of the laboratory oxidation tests, oxidation and condensation mainly occur, and the cracking plays a very insignificant part.

In Fig. 15 the graphs of the six determinations of the ageing from 100° up to 230° C. of the two lubricating oils *A* and *B* have been represented on a reduced scale in order to facilitate a comparison.

The following may be inferred regarding the ageing process :

Lubricating Oil A.

From 100° to 140° C.	Practically no ageing.
„ 140° „ 180° C.	Only very slight oxidation.
„ 180° „ 218° C.	Rapid beginning of oxidation which generates acids.
Above 193° C.	The acids begin to resinify (showing slighter increase in the acid number and rapid rise in the saponification number) whereas the conversion to, or formation of asphaltic matter may already have started. Pronounced condensation causing rapid increase in the viscosity, especially of the specific gravity and the Conradson residue.
From 218° to 230° C.	Progressive condensation (further increase in the specific gravity and the Conradson residue) (sludging). The sludge consists for the major part of asphaltic matter, formed at the expense of the acids and the petroleum resins in particular. For this reason the acid number and the saponification number above 218° C. change only a little. Owing to the sludging and the progressive condensation, the viscosity increases very rapidly.

Lubricating Oil B.

From 100° to 140° C.	Minor incipient oxidation (slight increase in the acid number).
„ 140° „ 193° C.	Mainly steadily increasing oxidation, condensation still playing a very minor part (slight increase in the viscosity and fairly slight increase in the specific gravity and Conradson residue).
„ 193° „ 230° C.	Progressive and still more pronounced oxidation (rather more rapid rise in the acid number), greater condensation (marked increase in the specific gravity and Conradson residue and increase in the viscosity).

The sludging which starts above 180° C. and is not serious up to 205° C. gathers momentum above 205° C. The sludge will probably be of different composition from that of lubricating oil *A*, whilst the sludge-free part of the oil may also be different.

This fact may explain why the increase in the viscosity above 205° C. is not very pronounced and why the acid number and saponification number keep increasing.

With the ageing in the oxidation instrument, designed by A. Mollinger, lubricating oil *A* is consequently stable up to 180° C. Marked progressive condensation and a moderate oxidation start above this temperature.

Explanation of Figs. 9-14.

—————	= Lubricating oil B without addition.
-----	= Lubricating oil B with 0.3% of very fine copper turnings.
⊙	= Lubricating oil B original before ageing.

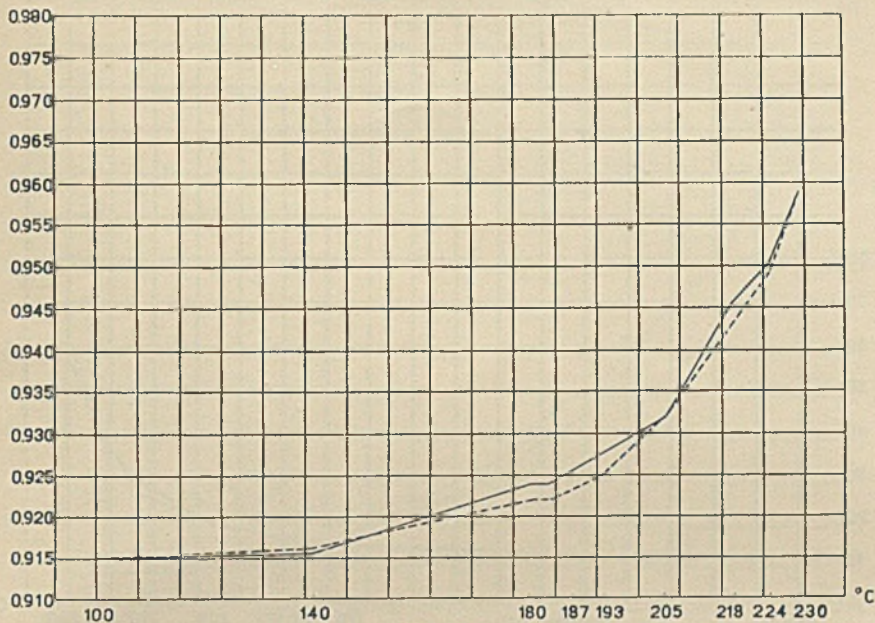


FIG. 9.

CHANGE IN SPECIFIC GRAVITY AFTER AGEING AT DIFFERENT TEMPERATURES.

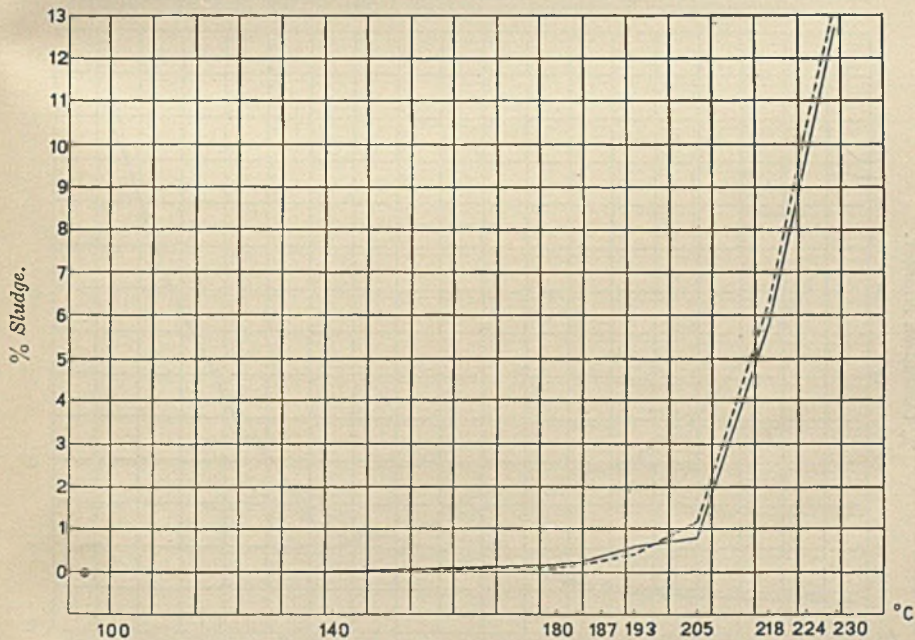


FIG. 10.

FORMATION OF SLUDGE (SOLUBLE IN BENZENE) AFTER AGEING AT DIFFERENT TEMPERATURES.

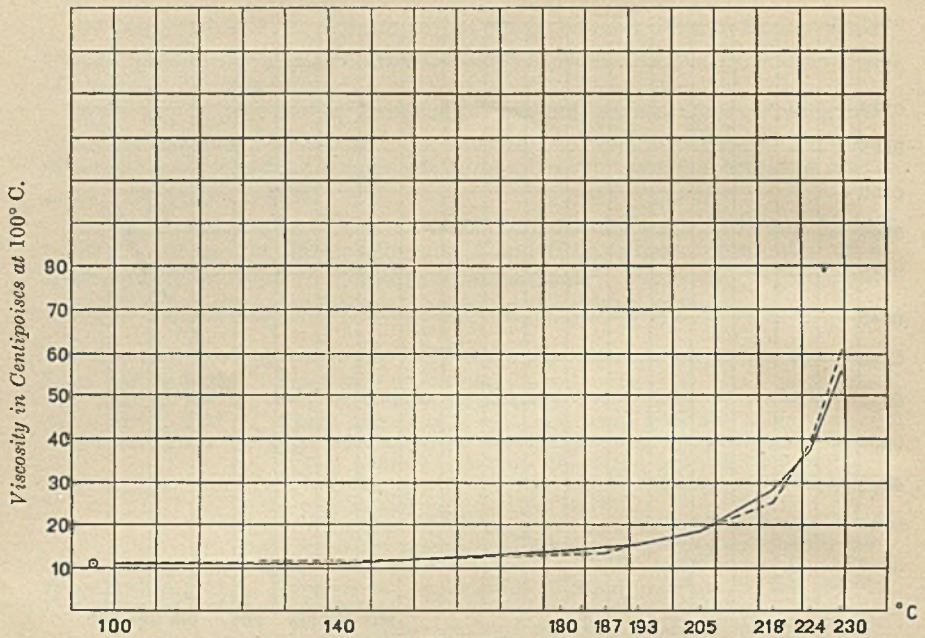


FIG. 11.

CHANGE IN VISCOSITY AT 100° C. AFTER AGEING AT DIFFERENT TEMPERATURES.

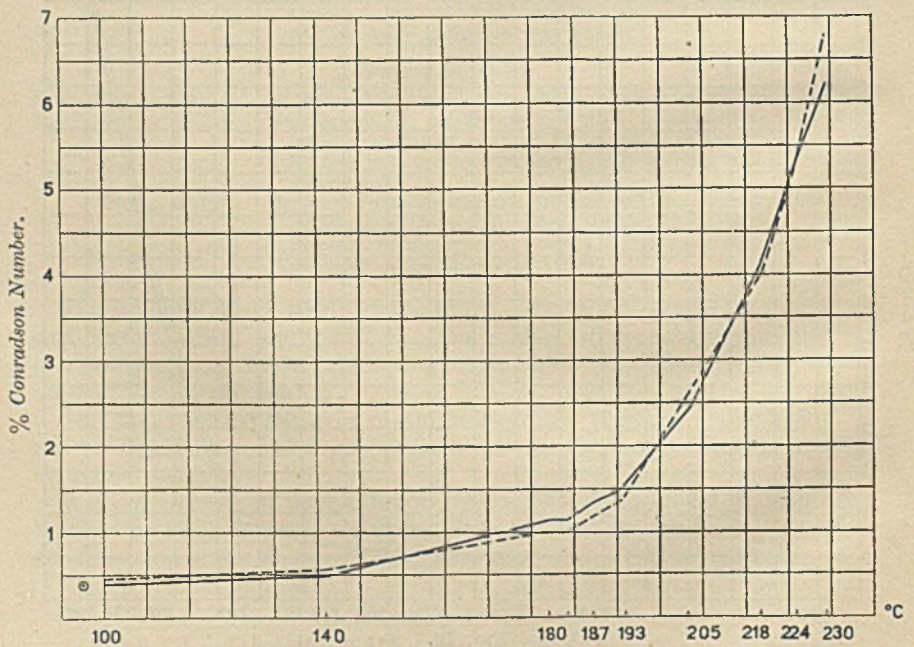


FIG. 12.

CHANGE IN CONRADSON CARBON RESIDUE AFTER AGEING AT DIFFERENT TEMPERATURES.

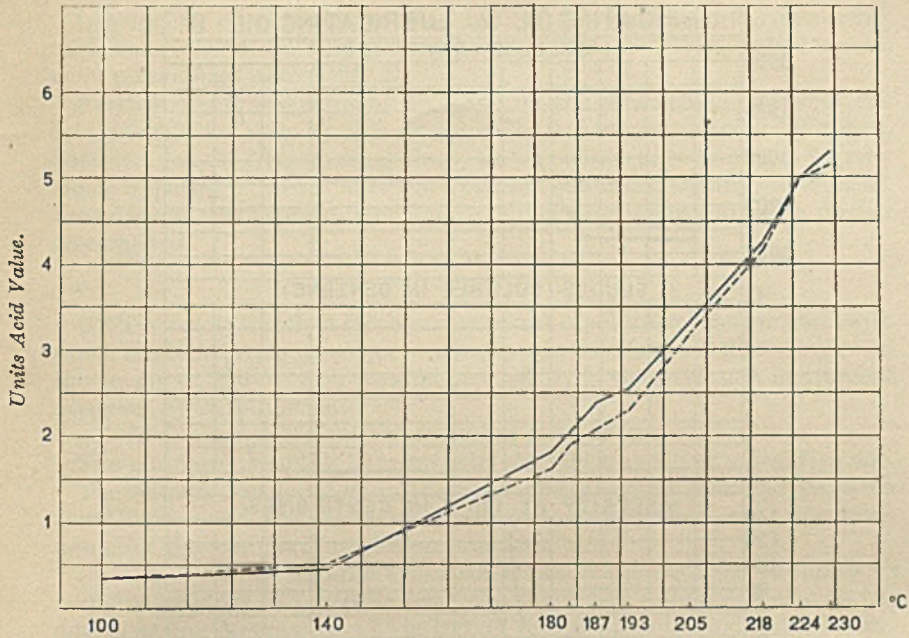


FIG. 13.

CHANGE IN ACID VALUE AFTER AGEING AT DIFFERENT TEMPERATURES.

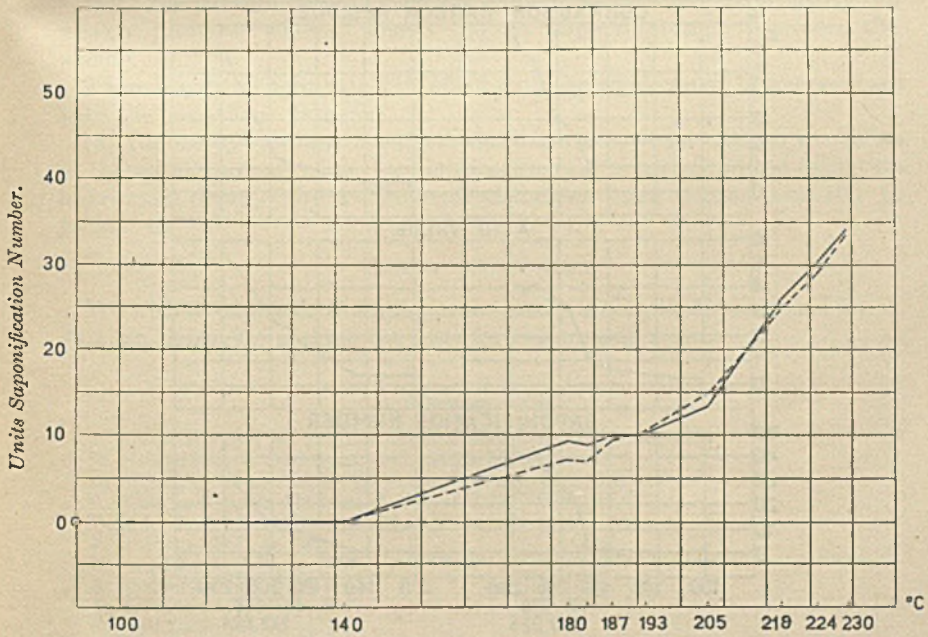


FIG. 14.

CHANGE IN SAPONIFICATION NUMBER AFTER AGEING AT DIFFERENT TEMPERATURES.

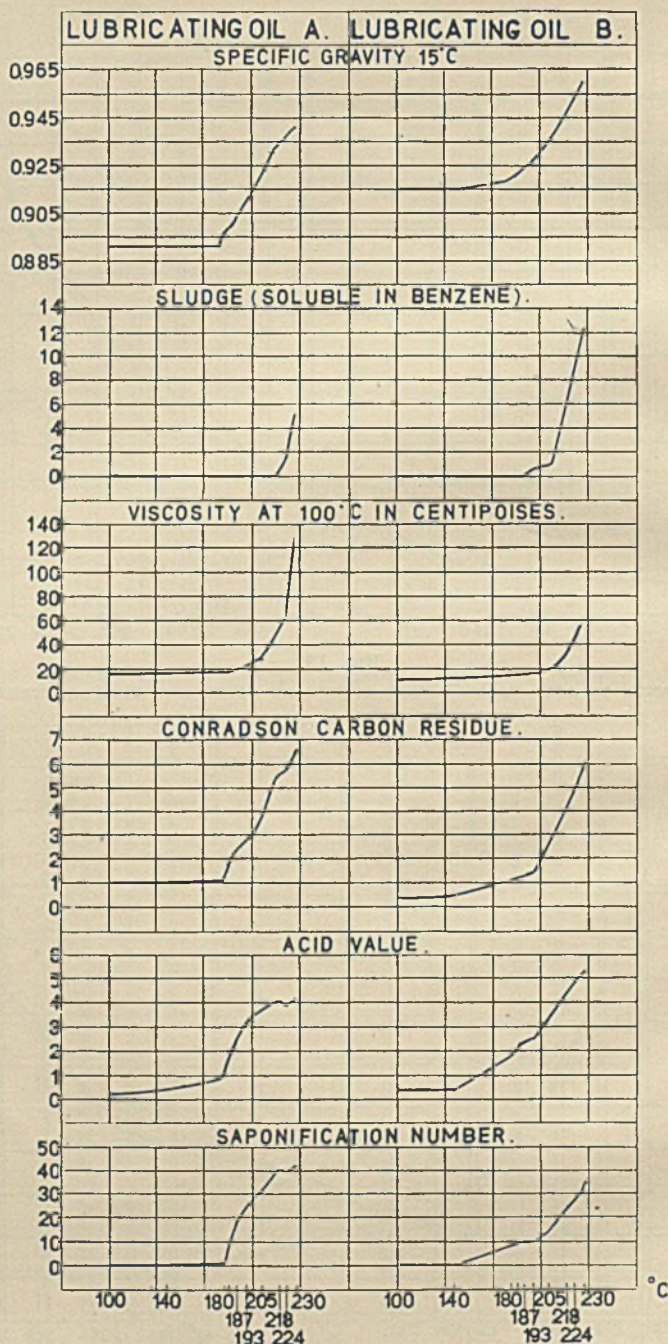


FIG. 15.

AFTER AGEING AT DIFFERENT TEMPERATURES.

Lubricating oil *B* is stable up to 140° C. Beyond this ever-increasing oxidation sets in, as does condensation, which, however, only becomes more pronounced above 193° C.

Further observations might be made on the remarkable course of the catalytic effect of copper on the ageing of the two lubricating oils. We shall, however, refrain from making them, as we should have to resort to very vague hypotheses, which have not yet been confirmed by any experiment. Nevertheless this problem seems to us sufficiently important to be further investigated.

SUMMARY.

(1) The catalytic effect has been examined of all the relevant metals with which the lubricating oil comes into contact in the Maybach diesel engine on the ageing at 230° C. of a paraffin base oil *A*, in the oxidation instrument designed by A. Mollinger.

All the ferrous metals stimulate ageing more or less.

The non-ferrous metals have no catalytic effect at all, or a negative one.

The minute iron splinters in the used lubricating oil from the Maybach diesel engine, and also the cylinder wall, piston-ring, piston, etc., will therefore alter the content of oxidation products.

(2) From the investigations into the effect of the addition of copper as very fine turnings to a paraffin base oil (*A*) and to a naphthene base oil (*B*) during ageing progressively from 100° C. to 230° C. in the oxidation instrument designed by A. Mollinger, it is evident that the catalytic effect of copper is very different in the case of the two lubricating oils.

With the paraffin base oil the catalytic effect up to 180° C. is clearly but weakly positive, whereas above 180° C. copper strongly represses the ageing.

There is only a slight catalytic effect over the whole range of temperature with the naphthene base oil.

(3) Guided by the data of the relevant literature, we have given an outline of the ageing process of the two lubricating oils (*A* and *B*) in the oxidation instrument designed by A. Mollinger at temperatures ranging from 100° C. to 230° C.

ACKNOWLEDGEMENT.

We wish to express our thanks to the analysts, Mr. R. Monster and Mr. J. van den Brink, for making the tests for this investigation.

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BURNING TEST FOR KEROSENE.*

I.P.T. Serial Designation—K 35 (T).

APPARATUS.

THE lamp to be used for this test shall consist of an oil chamber, a burner, and a chimney conforming substantially to the dimensions stated in Fig. 1.†

It is essential to ensure that the burner fits vertically into the lamp reservoir, and that the wick guide has parallel sides and is centrally disposed in relation to the slot in the dome of the burner. Any distortion of the wick guide and/or dome will make the production of the necessary flame shape difficult and render subsequent char values unreliable.

Wick.—The wick shall be a $\frac{3}{8}$ -in. (1.9-cm.) paraffin flat wick, super quality, as manufactured by Messrs. Morgan, Crossley & Co., Ltd.‡ It shall contain approximately 43 ends of 3-ply yarn, woven double plain weave with stitching ends, one blue stripe on one face and one green stripe on the reverse face: woven with approximately 15 picks per inch and weighing normally $\frac{1}{2}$ oz. per yard.

After weaving the wick shall be purified by boiling in distilled water and thoroughly dried.

After drying, the wick shall be made into rolls and left for 7 days before finally cutting into 8-inch (20.3-cm.) lengths. After cutting, the wick shall be packed into metal (tin) containers.

The maximum ash content of the wick shall be 0.4%.

Preparation of Apparatus.—New lamp glasses shall be soaked in diluted hydrochloric acid (1 : 1) for 24 hours, cleaned with a test-tube brush, and rinsed thoroughly with distilled water. The glasses shall then be subjected to three preliminary 24-hour burning periods before being used for burning tests.

After use, glasses shall be thoroughly cleaned, rinsed with distilled water and dried. Prior to each test the burner shall be thoroughly cleaned, removing any deposit from the wick guide, air holes and ducts. The oil container shall also be rinsed two or three times with a little of the oil to be tested. For each test a new piece of wick shall be dried in a steam oven for thirty minutes, soaked in the oil while still hot and fitted into the wick guide. The length of wick used shall be 8 in. (20.3 cm.).

The test shall be made in a well-ventilated room free from draughts.§

The minimum temperature of the room and the oil in the container at the start and during the test shall be 60° F. (15.5° C.). Lamps shall be so

* This method supersedes the method issued in 1938.

† Suitable lamps, burners and chimney glasses can be obtained from the Lamp Manufacturing and Railway Supplies, Ltd., River Plate House, 12/13, South Place, E.C.2, but they should be checked as regards dimensions before being used.

‡ Messrs. Morgan, Crossley & Co., Ltd., Ducio Mills, Manchester, 10.

§ If necessary, a suitable screen about 2 ft. (60 cm.) diameter should surround the lamp to ensure this.

BURNING TEST FOR KEROSENE.

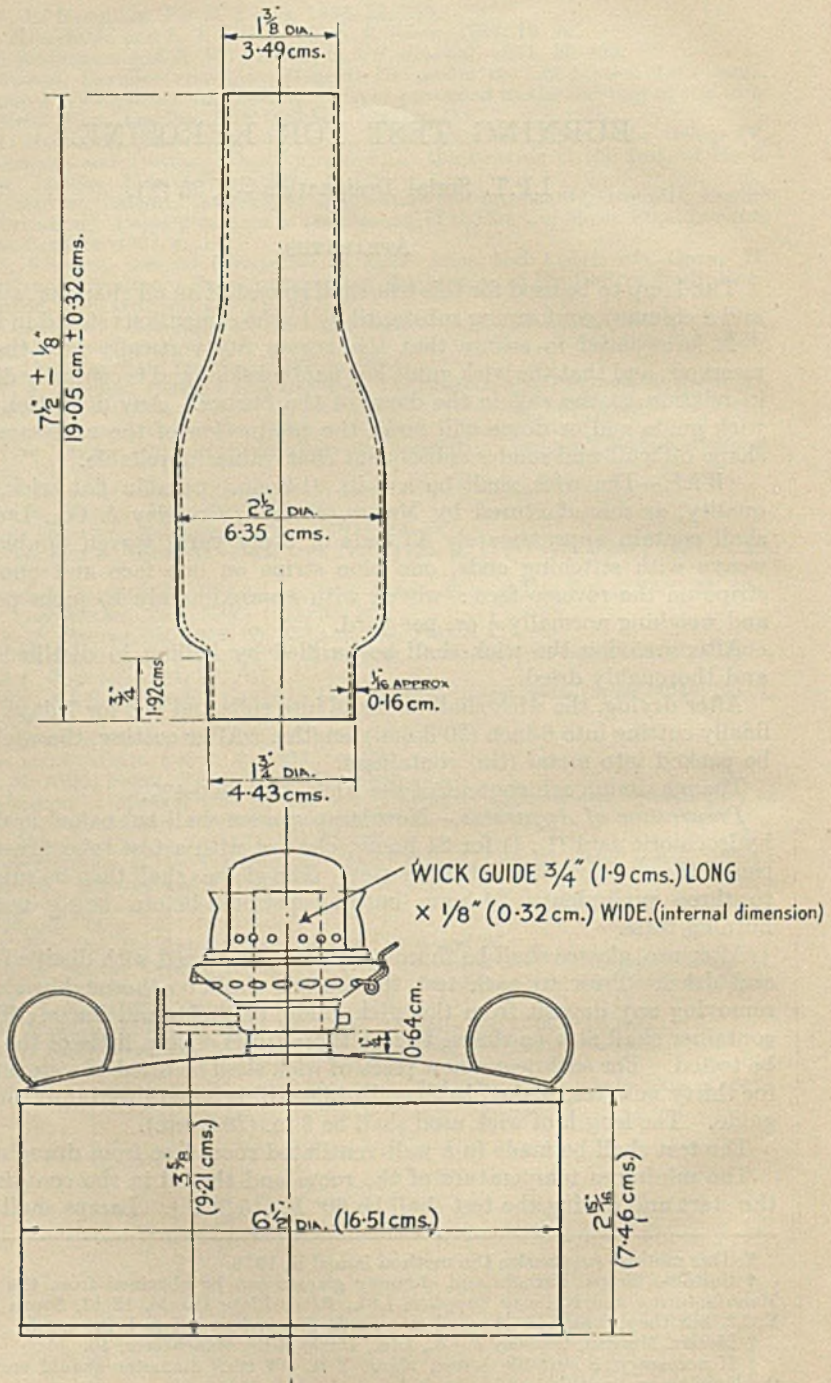


FIG. 1.

Note.—The tolerance on the chimney dimensions shall be $\pm \frac{1}{32}$ " except where otherwise stated.

placed as to be at least 1 ft. (30 cm.) apart and 1 ft. (30 cm.) from any wall.

DURATION OF TEST.

The test shall normally be of 24 hours duration, but in cases of dispute the duration of the test shall be 48 hours.

METHOD.

900 mls. of the oil shall be placed in the container,* and the burner, with the wick and clean chimney, fitted to the lamp. The dome (with chimney attached) shall then be folded back, and the wick carefully trimmed with sharp scissors to produce a flame 1 in. (2.5 cm.) high (measured from the top of the dome) and 1 in. (2.5 cm.) in width. The flame shall be smooth and symmetrical. Diagram A, Fig. 2, illustrates the flame shape with essential dimensions. It is to be emphasized that flame shape has a strong influence on char formation, and that it is therefore necessary to produce a flame similar, as nearly as possible, to that illustrated. The operation of wick trimming shall be carried out as follows:—

(a) The wick to be cut level with the wick guide.

(b) An inch or two (2.5 to 5.0 cm.) of the wick to be exposed and, starting at a point approximately $\frac{1}{8}$ in. (0.3 cm.) from each corner, a triangular portion to be cut off, as illustrated in Diagram B, Fig. 2.

(c) The sharp corners produced by operation (b) to be very slightly rounded off.

(d) Ragged projections, if any, to be removed by slightly bevelling the top edges of the wick illustrated in Diagram C, Fig. 2.

After trimming the top of the wick, it shall be re-saturated with kerosine before proceeding with the test. The lamp shall be weighed and the flame adjusted to within $\frac{1}{8}$ in. (0.15 cm.) of the standard flame dimensions.

The measurements of the correct height and width may be obtained by the use of the "Rather" Burning Test Gauge (see A.S.T.M. Serial Designation D-187/30), but other gauges, capable of measuring vertical and horizontal dimensions within the accuracy specified, are equally suitable. The gauge shall be placed as near as possible to the flame (say, 6 in. (15 cm.) from it) during this operation.

The lamp shall be allowed to burn for one hour, and the flame shall then be re-adjusted, if necessary, to the standard height. No further adjustments shall be made throughout the test.

Burning shall now be continued for a further 23 hours, and at the end of this period the lamp shall again be weighed.

In the case of the 48-hour test the lamp shall then be recharged with sufficient kerosine to restore the original weight, and burning shall be continued for a further period of 24 hours. At the completion of the test the lamp shall be again weighed.

The total consumption and average consumption per hour shall be

* If the previous examination of the sample had indicated the presence of suspended matter, the oil shall be filtered through a No. 4 Whatman (or equivalent) filter paper before carrying out the burning test.

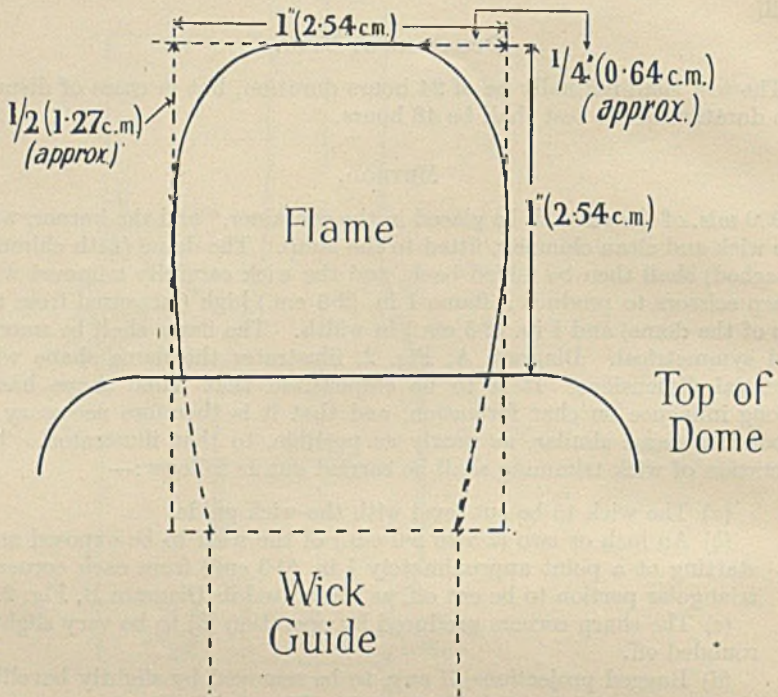
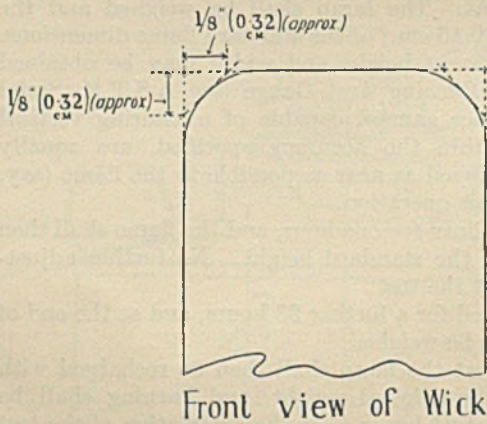
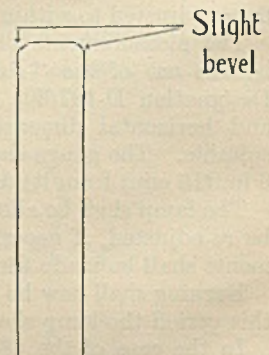


DIAGRAM A



Front view of Wick

DIAGRAM B



Side view of Wick

DIAGRAM C

FIG. 2.

recorded. (The average consumption will normally be approximately 20 grams per hour.)

Removal of Char.—A length of approximately $\frac{1}{2}$ in. (1.3 cm.) from the charred end of the wick shall be cut off and placed, with any pieces of char which have been detached during burning, or adhere to the wick guide (the char being removed by gentle scraping with metal forceps), in a small beaker and well washed by decantation with petroleum ether. The beaker and contents shall then be dried at 105/110° C. for half an hour.

After drying, the char shall be removed from the wick by—

(a) Breaking its structure by gentle pressure with metal forceps or similar instrument.

(b) Gentle scraping along and across the wick with the point of metal forceps or similar instrument.

Care shall be taken to ensure complete removal of char from the wick.

The mixture of char and fibre shall then be transferred to a glass plate or a sheet of glazed paper, pieces of thread from the wick shall be first removed, and the coarse char collected on a tared watch-glass. The fine, fluffy fibre shall then be removed as completely as possible from the mixture of fine char and fibre by tapping the mixture from one smooth surface to another, and gently brushing the mixture with a brush having short, stiff bristles. The object of brushing is to collect the fine fibre on the brush without removing the char. The mixture of fine char and fibre remaining on the glass or paper shall be finally brushed on to the watch-glass containing the coarse char, and the whole weighed. Results shall be expressed in milligrams of char per kilogram of kerosine, and duplicate results should be within the limits of $\pm 15\%$ or ± 2 milligrams, whichever of these is the larger.

RECORDING OF RESULTS.

The following shall be recorded :

Total consumption in grams.

Average consumption per hour in grams.

Weight of dry char per kilogram in mgms.

Bloom on glass :

(a) Predominating colour : Brown, greyish-brown or grey.

(b) General remarks : Normal or Unsatisfactory.

The maximum and minimum temperatures of the test room shall be recorded, and any abnormal atmospheric conditions noted.

THE INSTITUTE OF PETROLEUM.

LUNCHEON.

FRIDAY, 26TH JANUARY, 1940.

A LUNCHEON of the Institute of Petroleum was held on Friday, 26th January, 1940, at the rooms of the Royal Empire Society, Northumberland Avenue, London, W.C. 2 (by courtesy of the Council of the Society). This Luncheon was held in lieu of the Annual Dinner of the Institute, cancelled in December 1939.

The President (Professor A. W. NASH) presided, and among those who were present were :

The Rt. Hon. Lord Cadman, G.C.M.G., D.Sc., Colonel L. Pineau (Chairman, The Anglo-French Executive Committee for Oil), Sir Cecil Kisch, K.C.I.E., C.B. (Vice-Chairman, The Anglo-French Executive Committee for Oil), the Rt. Hon. Sir Ronald Graham, P.C., G.C.B., Sir Andrew Agnew, G.C.B. (Chairman, The Petroleum Board), Monsieur J. Filhol, Messieurs J. E. Bouchandhomme, M. E. Goldet, Maurice Mercier, Mr. K. L. Stock, Mr. E. E. Soubry, Mr. R. W. Sellers, Lt.-Col. S. J. M. Auld, M.C., O.B.E., Professor J. S. S. Brame, C.B.E., Mr. T. Dewhurst, Dr. A. E. Dunstan, Mr. J. Kewley, Dr. A. J. V. Underwood and Mr. F. A. Yearsley.

After the loyal toast had been honoured, Sir Andrew Agnew, G.C.B., proposed the Toast of

"THE PERMANENT ANGLO-FRENCH EXECUTIVE COMMITTEE FOR OIL"

He said : The great privilege has fallen to me of proposing the toast of the Anglo-French Petroleum Executive Committee. I took the trouble this morning to find out whether that was the proper name for that body, and I found that it was not. (*Laughter.*) The proper name, I understand, is "The Permanent Anglo-French Executive Committee for Oil." I think that the two things have much the same meaning, and I am sure you will all agree with me that they mean something very big indeed, something which the ordinary man in the street may not realize is a task of tremendous magnitude. It is a task of absolutely the foremost importance at a time like the present, when our minds, both in France and throughout the British Empire, are centred on one thing and one thing only, and that is this war and the winning of it. Petroleum is going to be one of the winning factors in this war, and the body that is going to be mainly concerned with that winning factor is this body whose toast I am now proposing.

Who are the people who are concerned with this body ? First of all, we have with us to-day, I am glad to say, the Chairman of the Committee, Colonel Pineau. (*Applause.*) We have with us also the Vice-Chairman, Sir Cecil Kisch. (*Applause.*) We have also with us M. Filhol and Sir Ronald Graham, but I am sorry to say that, owing to his indisposition, we have not with us Mr. Starling. They are the five—the Big Five—who are the members of this Committee.

It is almost unnecessary, perhaps, for me to say very much about Colonel Pineau. We ought to realize that Colonel Pineau, after his distinguished war service, has controlled the petroleum affairs of France since the last war, and I can assure you that all the energy, the drive, the knowledge, and the experience that he had at that time are still with him. He has not lost one single little bit of his energy; he has not lost one single little bit of his power of perception; and I know perfectly well that once again he is going to be one of the main factors in winning this war. (*Applause.*)



[To face p. 96.

LUNCHEON OF THE INSTITUTE OF PETROLEUM

London, 26th January, 1940.

The Vice-Chairman is Sir Cecil Kisch, a name which is perhaps not very familiar to some of you in connection with petroleum affairs; he has come comparatively recently within the scope of the trade. I am going very briefly to tell you something about him. He is a man to watch. He has brought into this business an amount of energy and drive and an amount of common sense that I can assure you are above the average, no matter where you go; so keep your eye on Sir Cecil Kisch.

M. Filhol, who is one of our guests from France, is a man who also has great experience in the petroleum world, and he is a man who will be of tremendous benefit to the Committee on which he sits. Sir Ronald Graham is a man of international repute, dealing with men and matters everywhere, and I am sure that he too will be of great benefit to the Committee. Mr. Starling we all know. Unfortunately, he is not able to be with us to-day. He is a man who has put in a tremendous amount of work.

This is the Committee of which I have to propose the toast. Could anyone wish for a nicer job than to propose a toast to these gentlemen, who are so well versed in the subject with which they have to deal, and so well known amongst us? I give you the toast with the very greatest confidence. I am glad to give it. I am glad to think that this Anglo-French Committee has been set up, and I am perfectly certain that it is composed of men who could not be bettered. I give you the toast of "The Permanent Anglo-French Executive Committee for Oil," and I couple with it the names of Colonel Pineau and Sir Cecil Kisch. (*Applause.*)

REPLIES TO TOAST.

Colonel L. PINEAU, who responded, said: This is the first time that I have had the honour of being your guest as the Chairman of the Anglo-French Executive Committee for Oil, but it is not the first time that I have had the great pleasure of taking luncheon or dinner with you. If this time the circumstances are more serious, and if the honour is greater, the pleasure remains the same.

More than ten years ago I was received as an Honorary Member of the Institution of Petroleum Technologists, which has since acquired titles of nobility and become the Institute of Petroleum. The Institute is the elder brother of the Association Française des Techniciens du Pétrole, whose affectionate greetings I am charged to convey to you, greetings of brothers in arms. (*Applause.*) I am able to tell you that, following your example, the "A.F.T.P." has decided to go on with its work. They will undertake again the editing of their *Bulletin*, and you will have an opportunity of following their work. You will also be able to find in this *Bulletin* news of many French engineers who have taken up arms and are at the front. (*Applause.*)

I have been your host as Chairman of the Permanent Council of World Petroleum Congresses, where I had as colleagues some very good friends like Colonel Auld and Mr. Dewhurst. I have an interesting communication to make to you on the subject of these Congresses. Through a neutral, the Chairman of the Deutsche Mineralölforschung conveyed to me that he is rather concerned about the Third Congress (*laughter*), which, as you know, was to take place in Berlin in June of this year. I was not very surprised. (*Laughter.*) The Germans, I am told, have made every effort to prepare for the Congress, but they desire that the Congress should keep its international character, and their acquaintances have grown fewer; and so they suggest that the Council in its entirety should meet in a neutral country. I wondered whether at the same time we should summon our Austrian, Czech, and Polish friends to the table of the crocodile mentioned by Mr. Winston Churchill! (*Laughter and applause.*)

Finally, I thought it would be better to excuse us for not going, and I answered that certain supplementary work having been thrown on us since September last (*laughter*), we, British and French, were compelled to devote the whole of our time to its execution until it has reached its completion and happy termination by victory. (*Laughter and applause.*)

It is with this aim in view that there has been created the Anglo-French Executive Committee for Oil, in whose name I have the honour of responding to this toast. To tell the truth, the creation of the Executive Committee has only sanctioned a collaboration between the two countries to which we have been accustomed for a very long time. I found again in the Executive Committee as technical adviser your

eminent Past-President and my old friend, Lord Cadman, whose name has been linked with all petroleum policy, whether national or international, for more than twenty years. (*Applause.*) I also found again in the Executive Committee Mr. Starling, with whom for a long time I have had, and still have, the great pleasure of working for Franco-British collaboration on questions of petroleum. Then I have found in the delegation appointed by the British Government newcomers of the first rank—Sir Ronald Graham, who has left on the shores of the Latin Mediterranean the fame of a great ambassador, and, above all, Sir Cecil Kisch, with whom we are on the best possible terms, and who has already become my friend. (*Applause.*)

The Executive Committee for Oil holds its meetings alternately in London and in Paris. This gives to its members the opportunity of making useful comparisons between the situation on the banks of the Thames and the situation on the banks of the Seine; to compare, for instance, the London blackout with the Paris blue-black-out. (*Laughter.*) One is no better than the other, and one gets sprained ankles in both cities! But one notices on both sides of the Channel the same fervour in doing one's duty, the same fierce resolution, the same certainty of victory. I do not believe that history ever recorded an alliance as complete and as close between great peoples engaged in the same cause. (*Applause.*) This cause is the cause of civilization as we have understood it for centuries, we British and French. If we believe Marshal Goering, Germany is to become only a factory, a barrack, a kitchen. Great Britain and France have another ideal! Our ideal is one will, one action, one victory. Let us work for it, and work efficiently, in the way the British so well know when the time comes. Let us follow the precept of the chronicler, at the same time an effective philosopher, when he said "Let us concentrate on action." In war, as in business, action is all that matters. Our principle here is "Let us talk if it is an aid to action, but, if it is an impediment or a substitute, let us keep a purposeful and forceful silence." This principle now invites me to hold my tongue and end my speech. (*Applause.*)

Sir CECIL KISCH, K.C.I.E., C.B., who also responded, said: It is difficult to follow Colonel Pineau, who has voiced so aptly the sentiments that I should like to express myself, but I do most honestly share his view on all the matters to which he has referred. Surely that is a good beginning for an Anglo-French co-operative executive!

I should like to thank Sir Andrew Agnew for the all-too-kindly references that he made to me personally, and to thank him in the name of the Joint Executive for what he said about their task. It is a formidable task that Sir Andrew expounded to you, and I can only say that when I was called into the Ministry of Mines my knees knocked, my octane number was definitely on the low side, and there was an urgent need for lead, but no one was likely to take the responsibility of administering it. I felt—to take the title of a book of which the title is appropriate—as though I was being plunged into Wells of Loneliness. (*Laughter.*) No lead there!

But what do I find around me? I find a host of friends, kindly, sympathetic, and generous, of which this gathering here is an abundant testimony. The Institute of Petroleum, our hosts, have a world-wide reputation. The members are men who have helped to make the industry what it is to-day by their scientific achievements in numerous directions. I understand that the year 1939 was a great year of achievement, but others to follow will be as great, and, I am convinced, greater. An avenue of unlimited promise lies ahead—an avenue "at whose immensity even soaring fancy staggers."

Turning from the general to the concrete, one knows that it is the work done in the science on which this industry depends that is giving our airmen this magnificent aviation spirit which will establish the Allied lead in the air. (*Cheers.*) This is just one illustration of the incalculable value of the Institute's work.

The Oil Executive has been broken in—no, I am using the wrong term; I should say it has been cracked in! (*Laughter.*) I understand—learning slowly, as I am, the special vocabulary of petroleum—that we have been polymerized; that is to say, we have been fused from a large number of units into one single, coherent unit. M. de Monzie, the French Minister for Public Works, who follows our work with the closest interest, observed that the Anglo-French Oil Executive had been fused by "un coup de foudre technique." That is a good start for harmonious work and good results.

The satisfactory functioning of the Executive, the Ministry of Mines, and all who are concerned with petroleum depends on the relations which exist with the great

industry inseparately associated with us in all our tasks. Our relations in time of war have to be characterized by a complete identity of objective and mutual reliance. We are trying to develop these relations in every way, and we had the advantage of the presence yesterday of representatives of the industry at a meeting of the Oil Executive. The Oil Executive and the industry must form one unit in carrying out the great task of which Sir Andrew Agnew spoke. We intend to expand this co-operation, now in full swing under most happy auspices. I may say that in a fairly long experience of public service I have never known such close combination and co-operation as exist to-day between the industry and the Government department which is its representative in the councils of the State. (*Cheers.*)

Petroleum is at the heart of our problems. None of you here will have any difficulty in answering the question, "Daddy, what did you do in the second great war?" You need only say, "I was engulfed in oil." That is at the bottom of everything, and everyone who is in it may fairly feel that he is doing his bit. In this work there are two great tasks before us. The first is to guarantee the large supplies of the products that the French and British require, and the second is the exclusion of those supplies from the enemy. In both those tasks the Direction des Carburants and the Ministry of Mines rely on the co-operation of the great oil industries in France and Britain, and we are receiving that co-operation in ample measure. I should like to add also, following Colonel Pineau, that we work under the guidance of an enthusiastic Minister, who watches our work with close attention.

I do not wish in the present context to mention many names, as my catalogue of friends and colleagues in the work would be too long. Everyone knows, however, that this association with the industry is very close through the work of the Petroleum Board, whose Chairman has just spoken. A long time ago the poet Byron—who was also an expert on oil, though you may not have known or remembered it—wrote:

"In virtues nothing earthly can surpass her
Save thine incomparable oil, Maecassar!"

Byron was no doubt thinking of the beauty parlour and of the poet's locks; to-day the lines would be a little different, and I suggest that Byron might have written:

"Unearthly virtues fill thy spirit cool,
Hail, oil incomparable, tetra-ethyl Pool!"

(*Laughter.*) Sir Andrew Agnew wonders whether I am joking. I am not. I have not licensed my car this quarter, and I offer him the lines to advertise his spirit, free of all royalty or commission!

I must say one word to our Chairman, and, with your permission, I will return the compliment which he paid us by speaking in English and make those remarks in French. (*Continuing in French.*) I wish to take this opportunity to express my lively thanks for the very great skill and knowledge of Colonel Pineau, the Chairman of the Committee. All those who take part in the deliberations of the Committee and who are happy to work under his chairmanship are glad to pay a sincere tribute to the devotion with which he carries out the important tasks which have to be performed during the present critical period.

Sir Andrew has told you something about some of the other members, and I shall not go over the ground again, except to express my regret that Mr. Starling, suffering from the fashionable illness of influenza, is unable to be here, and has asked me to express his regret that he could not be present on this interesting and important occasion. We are glad to have found two admirable bilingual Secretaries—Siamese twins, two hearts that beat as one—who keep the minutes and the members in order with equal facility.

I must now refer to one whose name has already been mentioned, and who has come back to this work—Lord Cadman, who was at the Ministry before, in the last war. Lord Cadman, I am glad to say, has the room next door to mine, so that there is someone who can keep an eye on me—though I do not altogether accept Sir Andrew's imputation! Lord Cadman brings to our work wise counsel, long experience, and understanding of men and matters which are invaluable at a time like this, when so much depends on knowledge and so much depends on the capacity to handle men. I should like to express to him my personal acknowledgements.

All speeches must be rationed in these days, and I think that I have exhausted even my supplementary coupon. (*Laughter.*) But, rationing or no rationing, there

is always one kind of oratory that is satisfactory, and that is finished oratory (*laughter*), and so I should like to conclude by thanking Professor Nash and all you gentlemen who have so generously honoured us on this occasion for your kindness in receiving us, and by saying to our French friends who are here that to-day *Union Sacrée* has a wider meaning, and it is a *Union Sacrée* for us all until we achieve the final goal, victory and peace. (*Applause.*)

The PRESIDENT (Prof. A. W. NASH) said: It is very gratifying to me to-day to see such good support from the members of the Institute at this function. Our greatest pleasure has been to welcome the members of the Anglo-French Executive Committee for Oil and wish them success in their extremely responsible task. Another pleasure in which I feel we all share is that of renewing old friendships and acquaintanceships among our fellow-members; and in that respect particularly we are pleased to welcome our friends Colonel Pineau and M. Filhol, gentlemen whose scientific and technical knowledge we hold in the highest esteem, based as it is on a sound background of petroleum technology and a knowledge of the industrial problems connected therewith, a knowledge which, I should imagine, is most essential in work of the kind on which our French colleagues are now engaged.

This is an opportunity to meet which comes to some of us only on the occasion of the Annual Dinner, and, as you all know, it was deemed advisable to cancel the arrangements for the Annual Dinner, which was to have been held last December. You have also had to forego the usual monthly meetings of the Institute and the advantages of consulting the Institute's library. I feel sure you will allow me to trespass on your time for a few moments, while I give you a little more information than it has been possible to give in printed circulars.

A few days after the war broke out we were compelled by circumstances over which we had no control to vacate our London offices. In the few days we had available, our Secretary lost no time and spared no energy in endeavouring to get other accommodation in London or its outskirts, but without success. Fortunately there was a large room available at the Department of Oil Engineering and Refining at Birmingham University, and I was able to place that room at the disposal of your Council. The Council accepted that offer, and, as you all know, the Institute has been located at Birmingham since last September.

Apart from the fact that the whole of the Institute's business has now to be transacted through the post, the Institute's activities are no more and no less than they would have been if it still retained its offices in London. In the matter of monthly meetings, for example, the Publication Committee had prepared a very interesting and important syllabus of papers for the 1939-40 session, but without exception the authors of those projected papers were transferred to work of great national importance and were compelled to withdraw their promises. On the other hand, the *Journal* has continued publication without intermission, and, so far as I can say at present, will continue to do so. It may be of interest to you to learn that, in spite of many inevitable restrictions and excluding enemy countries, between two and three thousand copies of the *Journal* are posted every month all over the world.

Yesterday your Secretary and I attended a luncheon of the flourishing Northern Branch of the Institute at the Engineers' Club in Manchester, when I addressed our northern members on the subject of aviation fuels and lubricants.

In our period of exile from London, very fortunately we have found many good friends, and to all of them I should now like to tender our most grateful thanks. The Institution of Chemical Engineers have allowed us to use their premises for meetings of the Council and for Committee meetings. The Royal Empire Society have graciously allowed us the use of this very delightful dining-room for the present luncheon. The Institution of Mechanical Engineers and the Imperial College have given us the use of their libraries. The Institute of Fuel have invited our members to their meetings, and finally the Royal Society of Arts have allowed us to hold a meeting this afternoon in their somewhat war-scarred meeting hall—you will understand that statement when you get there—where I hope in a few minutes to have the pleasure of addressing you again. In the meantime, I ask you to show your appreciation of these very real kindnesses and tokens of help with acclamation.

The vote of thanks was carried by acclamation, and the proceedings terminated.

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Geology.

208.* Correlation of Surface and Subsurface Formations in Two Typical Sections of the Gulf Coast of Texas. A. Deussen and K. D. Owen. *Bull. Amer. Ass. Petrol. Geol.*, 1939, 23, 1603-1634.—From data now available, two sections of the Gulf Coast have been prepared giving details of the strata to a depth of 12,000 ft. The first, or East section, extends from Fayette Co. to Matagorda Co., and the second, or West section, from McMullen Co. to Nueces.

East Section.—The lowermost beds of this section belong to the Yegua formation, at the top being the Cockfield member. The Yegua is primarily a continental deposit with sands, peats, and clays of lake origin. These deposits, however, grade downward into a series of shales, clays, and sands of marine origin. Indicative of marine conditions is the presence of *Discorbis yeguacensis* which occurs in a well section at the top of the Yegua at a depth of approximately 3500 ft.

Above the Yegua follows the Jackson formation, which consists mainly of sands and sandstones with interbedded clays and shales. Casts of marine fossils occur in the sandstones. *Textularia hockleyensis* is present in all the well sections with the exception of the well nearest the outcrop, this occupying a definite and persistent plane in the formation 200 ft. below the top.

Overlying unconformably the Jackson occur Oligocene sediments made up of the Catahoula (sands, clays, fossil wood, and volcanic ash). This is overlain, also unconformably, by the Oakville sandstone, a formation containing vertebrate fossils of Miocene age. Between these two formations there is encountered at depth a wedge-shaped deposit consisting of a marine shale and a sandy series ("Frio Sand"), followed by another bed of shale, the Vicksburg.

The remaining and uppermost sediments comprise Post-Oligocene beds, represented by the Fleming series, and composed of clays and shales deposited in lakes and fresh-water lagoons.

West Section.—The same depositional sequence is recorded here as in the East Section. Certain differences in thickness and positions, however, are noted. Only the western attenuated edge of the shale wedge occurs, and there is no thickness of this shale comparable with that of the East section. Its corresponding thickness in the West section is probably farther in the basin and beyond the present coast-line. The sands below the shale (the so-called subsurface "Frio") reach a much greater thickness in this area as compared with that of the East section, and, moreover, they crop out at the surface between the Jackson and Catahoula.

Though the correlation of the Frio and Catahoula has for long been a debatable question of Gulf Coast geology, the authors state that evidence now points to the surface Frio as the outcrop time equivalent of the Vicksburg, both formations having been deposited under different conditions, the Frio in salt-water bays, and the Vicksburg in the open sea. It also seems that the Catahoula is probably the surface time equivalent of the marine Oligocene group, including the lower sands (subsurface "Frio"), the intermediate marine shale wedge, and the overlying sand series.

Finally, it appears that in the subsurface there are at least four definite stratigraphic units which can be mapped by the usual methods of subsurface mapping, now unnamed or loosely or improperly named.

G. S. S.

209.* Amelia Oil-field, Jefferson County, Texas. E. J. Hamner. *Bull. Amer. Ass. Petrol. Geol.*, 1939, 23, 1635-1665.—This field was discovered in 1936, and is situated within the salt-dome province of the Gulf Coast in Jefferson County.

Stratigraphically, the following formations and groups are present: Pleistocene (Beaumont clay), Pleistocene and Pliocene (Lissie-Citronello group), Pliocene and Miocene (Lagarto clay), Miocene (Oakville-Catahoula), Oligocene (*Discorbis*, *Heterostegina*, *Marginulina* zones, and the Frio formation). The Frio is at least 1800 ft. thick, and is the deepest formation yet penetrated at Amelia. It is made up of numerous beds of porous grey sands of varying texture. Nine distinct sand members are recognized, ranging from 25 to 475 ft. thick; these, with the exception of the top member, are fairly continuous and regular across the entire field. The Langham sand, the sixth member of the series, is the producing zone from 6694 to 6785 ft., and is the thickest (425 to 475 ft.) and best-developed sand-body of the group. An important feature of all the sands is their content of volcanic ash.

Structurally, the Amelia field is an elliptical flat dome adjacent to a big fault. The long axis of the structure trends north-east and south-west although the field is fairly symmetrical, the short axis of the dome is rather closer to the south-west end of the producing area. It is one of the first examples of an exceptional occurrence of production in the Gulf Coast, wherein the accumulation of oil lies on the downthrown side of the big fault rather than on the upthrown side.

The proved area of the field is 1180 acres, and to Jan. 1, 1939, the field has produced 2,644,642 bbls. of oil from 114 wells, giving an average of 2168 bbls. per acre. The Mary Langham No. 1 is the oldest producing well, and has yielded 77,000 bbls., with prospects of good future production.

Concluding sections of this paper deal with the development of the field, in particular with well-completion problems and methods. A new method of completing low gas-oil wells by cement squeezing was introduced at Amelia with very successful results, and it is now used in all doubtful wells before original well completion. A description of the method is given with a sketch showing how the squeeze job is carried out.

G. S. S.

210.* Standard Permian Section of North America. J. E. Adams *et al.* *Bull. Amer. Ass. Petrol. Geol.*, 1939, 23, 1673-1681.—The authors advocate the classification of the American Permian as a system of four series. These, in ascending order, are: the Wolfcamp series, Leonard series, Guadalupe series, and Ochoa series. This classification is based on the thick and nearly continuous section exposed in and immediately adjacent to the Delaware basin.

Wolfcamp Series.—This, the lowest division, includes the oldest Permian rocks of the Glass Mountains region, which here consists of about 600 ft. of limestones, conglomerates, and shales.

Leonard Series.—The second division comprises beds formerly assigned to the Leonard formation, which at the type locality (south face of the Glass Mountains) is more than 1800 ft., and consists of limestones and dark siliceous shales. It rests unconformably on the Wolfcamp.

Guadalupe Series.—This series comprises beds that have been classed as the Guadalupe group. At the type locality, the south end of the Guadalupe Mountains, the series is 4100 ft. thick, made up of 2300 ft. of sandstones overlain by 1800 ft. of (Capitan) limestone. In the Delaware basin the Guadalupe is represented by a sandstone facies—the Delaware Mountain sandstone, 3000-3500 ft. thick.

Ochoa Series.—This, the top division, is designed to include all upper Permian sediments of post-Guadalupe age. They consist mainly of evaporites having a thickness of considerably over 4000 ft. Four distinct subdivisions are recognized.

A brief reference is made to a suggested correlation of these series with beds in other areas beyond the limits of the Delaware basin.

G. S. S.

211.* Subsurface Cross-section of Permian from Texas to Nebraska. C. L. Mohr. *Bull. Amer. Ass. Petrol. Geol.*, 1939, 23, 1694-1711.—This paper has for its object the presentation of the regional stratigraphy of the "Blaine" and older Permian beds. In illustration of this, sample logs have been selected from twenty wells for a cross-section from Texas to Nebraska. The locations of the wells are shown on an index map. The section is not carried to the surface, but the supposed surface correlations and the nomenclature are discussed. As a standard for reference, the marine Permian section of the Glass Mountains is utilized, which is 150 miles south-west of the south end of the cross-section.

The principal divisions of the Permian represented are the Wolfcamp, Leonard, and the lower part of the Word of the Glass Mountain section.

Paleontological evidence shows that several separate and distinct faunas are related to the correlation and classification of the Permian, both in wells and on the outcrops. These faunas may be designated in ascending order as the Shawnee (or Graham), Wabaunsee (or Thrifty), Wolfcamp, Leonard, lower and upper Word, Capitan, Custer (?Permian), and Moenkopi (Lower Trias). These zones are nearly all bounded by regional disconformities which are fundamental in the scheme of the present cross-section.

Two major disconformities can be followed for the whole distance, one at the base of the Wolfcamp and the other at the base of the Leonard. A regional unconformity

occurs between the Dog Creek shale and the overlying Custer sandstones, and, in some wells east of the cross-section, the base of the Custer rests on the base of the Blaine.

The unconformity at the base of the Wolfcamp is correlated with the middle of the Harpersville in Central Texas, the base of the Wanette in Southern Oklahoma, and the base of the Admire in Kansas and Nebraska. The Leonard-Wolfcamp unconformity probably lies just below the Elm Creek limestone in Central Texas, at the top of the Wanette in Central Oklahoma, and just below the Herington limestone in Kansas and Nebraska.

Figures of cross-sections of the Permian from Texas to Nebraska and from Texas to Oklahoma are furnished, together with a regional correlation chart. G. S. S.

212.* Oil Occurrences in South-West Lancashire. F. W. Cope. *Bull. Geol. Surv. Gt. Brit.*, No. 2, 1939, 18-25.—During a re-survey of the Formby District (S.W. Lancs.) seepages of oil were found in, and close to, Downholland Moss Lane, about a mile east of Formby. Indications of oil were also noticed at Barton Moss and Flea Moss, about two miles east and south of Formby, respectively.

At Thirty Acre Lane, close to the junction of Downholland Moss Lane, the occurrence of the oil is most striking, in that it is an active seepage. Preliminary examination of neighbouring drainage ditches cut in peat led the author to suspect that the oil originated in the peat. Later, an auger was used to investigate the occurrence, and sampling was carried out to a depth of about 16 ft. The section showed alternating beds of peat and silty clay, the peat containing much oil. During augering a good deal of gas was given off, which readily ignited.

A series of laboratory tests made by the Anglo-American Oil Co. shows the oil to have a specific gravity at 60° F. of 0.8885 and a low saponification value, the latter probably indicating a mineral origin. Examination of oil extracted from a peat sample taken near this section, however, appeared to be of vegetable origin. Thus, two types of oil were detected.

From other localities in the Downholland Moss oily peat and drops of oil were seen in ditches on either side of the Lane.

About a third of a mile to the north a shaft was sunk to a depth of 22 ft. in the bank of Downholland Brook and a similar section to that at Thirty Acre Lane was revealed. Oil was plentiful in the upper peat bed and also in the underlying silty clay. Considerable volumes of gas were liberated, and 7-8 gals. of oil accumulated in the shaft during one night.

Conclusions strongly favour a deep source for the oil. (The type obtained from the peat probably originated from a small quantity of oil-producing algae present in this material.) It is tentatively suggested that most of the oil may be derived from a buried Carboniferous structure beneath the Trias, and that it reaches the surface along the Hillhouse Fault. Little is known, however, of the thickness of the Trias in this area, and there is no evidence as to the character of the underlying Carboniferous. Further details of these rocks and more definite information regarding the South-West Lancashire oil occurrence will doubtless be available when exploratory boreholes are sunk in the Formby area.

At the end of the paper a biological report on the peat is given by Dr. K. B. Blackburn. G. S. S.

213.* Exploratory Boreholes. L. Migaux. *Bull. Ass. franç. Tech. Pétrol.*, 1.7.39 (50), 5-34.—An exploratory well must give the fullest possible information on stratigraphy, structure, and the nature and content of the permeable horizons. Rotary drilling is now used almost exclusively for this purpose, and the tendency is for drilling depths to increase and equipment to become heavier. Stratigraphical evidence is obtained from fossils, generally micro-organisms which require very detailed study, lithology, and the physical properties of the rocks (elasticity, electrical, etc.). Evidence obtained from the mud flush is very uncertain, especially as regards information on depth, and mechanical coring is preferable. Even this, however, is not perfect, since the core recovery is only 65-90% and the parts lost are the more porous and friable rocks which actually are the most interesting. A record of drilling speeds is useful in some areas. Electrical coring is almost universally used, and this, in conjunction with the above methods and side-wall sampling, will give sufficient information.

Structural evidence may be obtained from cores, although allowance must be made for deviation of the bore-hole from the vertical. This deviation may be measured, but the orientation of a mechanical core is still unknown. Hence one is dependent finally on some method such as the Schlumberger dip meter or by magnetic measurements on the core itself.

The presence and position of permeable horizons are given by electrical coring. The simplest method of testing these horizons would be to take side-wall samples after drilling was complete. At present, however, it is general practice to core mechanically in zones thought to be interesting. Mud flush samples and the electrical log assist in selecting such zones. Examination of these cores will indicate the type of fluid present in the permeable beds, but production tests are necessary to get full details of their probable value. Production tests may be carried out in various ways, but the best methods are those which test individual horizons. For this purpose a formation tester may be used, or tubing may be cemented at the base of the zone to be tested and then perforated at regular intervals above, tests being made after each perforation and the holes re-cemented before the next perforation is made. S. E. C.

214.* Discoveries Incite Vigorous Exploring on Four Mississippi Fronts. G. Weber. *Oil Gas J.*, 2.11.39, 38 (25), 13-15.—Interest in Mississippi is concentrated on four fronts—Tinsley field, Yazoo County; Vicksburg-Jackson district; Mississippi Sound; north-east Mississippi (including north-west Alabama). Over 1,530,000 acres were under lease prior to the Tinsley discovery.

The Tinsley field has shown a second oil zone under the discovery zone. Wells have been completed south and north-east of the discovery well, and a well to the north missed the Woodruff sand, but cored a good oil section in the deeper Eutaw formation at 2900 ft. Coring below 5040 ft. gave no other shows.

On the Vicksburg-Jackson trend the Glass dome and Blakely prospect of Warren County and the Sartartia structure near Tinsley are of interest. This area has sub-surface formations similar to those of North Louisiana and Arkansas, and is somewhat faulted.

Ten gravity-meter, ten seismic reflection, two magnetometer parties, and one soil analysis party are at work in Mississippi. G. D. H.

215.* Nebraska Added to List of Oil-producing States. D. Dalrymple. *Oil Gas J.*, 9.11.39, 38 (26), 9, 10.—Commercial oil has been found in the Forest City basin of South-east Nebraska. The productive Hunton lime was met at 2288 ft. Production is estimated at 75-200 brl./day of 30° oil. The oil shows very little gas. The well-site was chosen after seismic work. In 1937 a well drilled 10 ml. to the west found some oil showings, but it was not possible to shut off the water. This test found the Mississippian, Silurian, and Devonian over the Viola relatively thin. Other wildcats have had oil showings. G. D. H.

216.* More Tests Follow Strike in Nebraska. D. Dalrymple. *Oil Gas J.*, 17.11.39, 38 (27), 89.—The bulk of drilling activity in the Falls City basin is in the Missouri sector. Several dry holes have been completed, but some new gas areas have been reported. Operations are also expected to increase in North-east Kansas following the Falls City strike. The Falls City discovery well found the Hunton lime at 2276 ft., oil at 2279 ft., and was bottomed at 2282 ft. On deepening to 2287 ft. more oil saturation was found. G. D. H.

217.* Wildcats, Oil Shows Accelerate Interest in Mississippi. Anon. *Oil Gas J.*, 17.11.39, 38 (27), 92.—Activity is centred on the Vicksburg-Jackson area, the interest being shared by the North-west Alabama basin which extends into North-east Mississippi. Oil seepages have been reported in Wilkinson County, and oil and gas shows in wildcats in Itawamba and Kempen Counties.

The Tinsley field is giving 325 brl./day. A sand at 5493-5508 ft. on the northern edge of the field has given salt water instead of being the hoped-for third oil sand. The Eutaw at 4865-4877 ft. has shown oil and is below the Selma section, which is producing in the present three wells.

In North-east Mississippi a test has reported a showing of oil or gas at 560-570 ft. in hard lime.
G. D. H.

218.* Development of Two New Producing Zones Faces Salem, Illinois, Operators. H. F. Simons. *Oil Gas J.*, 23.11.39, 38 (28), 8, 9, 98.—Prolific production has been obtained in a deep Devonian test at the northern end of the Salem field. This is the fourth oil zone tapped since the field's discovery, and the presence of a fifth major producing zone is indicated by the efforts to recomplete in the St. Louis lime two former McClosky producers.

The recent Sandoval, Bartelso, and Salem discoveries in the Devonian have encouraged search for this horizon elsewhere, and tests have been started in the Centralia townsite and North Aden pools. Between the McClosky and the Devonian pay is an almost complete lime section with few shale zones. This will increase drilling difficulties. The Devonian lime is very porous and highly permeable.

McClosky wells decline slowly to a certain point while flowing and then suddenly cease to flow. On turning over to pumping they yield but a small percentage of their flowing production.
G. D. H.

219.* Continental No. 1 Proctor is the Deepest Test in Mid-Continent Area to Date. Anon. *Oil Gas J.*, 23.11.39, 38 (28), 10, 11.—The well was taken to 14,479 ft. without any serious difficulties, but a series of fishing jobs ensued, and finally, at a total depth of 14,582 ft., it was decided to go back to test upper formations, amongst which a gas-bearing zone at 10,898 ft. was of special interest. This well was intended primarily to give information on the central part of the Anadarko basin of Western Oklahoma. It has shown that the thickness of Pennsylvanian beds in this basin is greater than was expected by most geologists. It has also revealed the value of diesel-electric equipment for deep drilling.
G. D. H.

220.* Mississippi Play is Extending Throughout the State. G. Weber. *Oil Gas J.*, 23.11.39, 38 (28), 25.—It is estimated that 5,000,000 acres are now under lease in Mississippi, almost twice the area leased before the Tinsley discovery. In Clarke County in 1931 a well showed heavy oil in the Eutaw at 3692-3700 ft. before being abandoned at 4775 ft. A new test is to be drilled near this. Other tests are projected or under way in Attala, Copiah, Harrison, Hinds, Issequena, Itawamba, Lamar, Monroe, Pike, Prentiss, Simpson, and Yazoo counties.

In the Tinsley field a well on the west side has failed to find the Selma chalk, but is coring in the Eutaw section. A well in the north is coring below 6000 ft. without having found any shows.
G. D. H.

221.* Mississippi is Experiencing a Real Oil Boom. B. Mills. *Oil Wkly*, 13.11.39, 95 (10), 42-53.—A ten-year search in Mississippi has revealed only one oil-field. The present boom is centred on the Tinsley discovery in Yazoo County. This involves Cretaceous beds, but the Palæozoic area in the north of the State also offers distinct possibilities at reasonable depths. The Amory gas-field is in the latter area. The southern part of Mississippi offers promise as a salt dome district, and the Cretaceous is at reasonable depths all the way to the coast. However, it is not present over all the State, and it is at greater depths in the west and south-west.

The Hartelle sandstone of the Upper Mississippian is perhaps the primary Palæozoic objective, for it has asphalt and oil along its outcrops in North Alabama. Many structures have been mapped, and gas has been developed at Fayette (Alabama) and Amory (Mississippi). There have been many oil-shows, but no commercial production.

Igneous intrusions are common in the Cretaceous belt centred near Yazoo City. The Tinsley dome has an uplift of 125 ft. at the surface and about 600 ft. of closure on top of the Selma Chalk (equivalent to the Annona Chalk of Louisiana and Arkansas). The oil has little gas in solution, and practically no gas is produced with the oil, which is asphaltic and of 34° gravity. It is not known whether Tinsley is a deep-seated salt dome or a true anticlinal structure. A 40-acre spacing and a 2000/1 gas-oil ratio have been enforced.

The Glass dome and the Blakely prospect in Warren County are due for tests, and there are other attractive structures.
G. D. H.

222.* **Nebraska and Forest City Basin Produce First Oil.** F. B. Taylor. *Oil Wkly*, 13.11.39, 95 (10), 72-76.—Oil has been found by a rank wildcat in extreme South-east Nebraska. A 24-hour pumping test gave 230 brl. of oil. The productive Hunton lime was topped at 2276 ft. and was drilled to 2282 ft. Forty-seven dry holes have been drilled in the Forest City basin. The main geological feature of the area is the Nemaha granite ridge, which runs approximately north-south. The strata dip abruptly from the ridge, and to the east of the well the sediments slope off gradually into the Forest City basin. A number of surface structures are known, but drilling has only given shows of dead, heavy oil. East of the ridge, and well into the basin, there has been seismic work and core-drilling. Deep-seated anticlines have been located, and a north-south trend is thought to have been established, but these more pronounced features have not been drilled. The Viola and Hunton limes are thought to be more likely to have commercial oil than the deeper Wilcox or Simpson.

G. D. H.

223.* **Arabia, Vital Crude Reserve of Near East, has Still Brighter Future in Oil.** C. R. Spencer. *Oil Wkly*, 4.12.39, 95 (13), 16-20, 22.—The first concession was granted in 1933 for an area bordering the Persian Gulf. In 1937 geophysical work was begun, and in 1938 core-drilling.

The general conditions in Arabia with regard to life, labour, and other features are discussed.

Production in 1939 was 275,000 brl., whilst the potential was 900,000 brl. Some sixteen wells were producing, and seven were being drilled in October 1939. A 10-inch pipe-line has been built from Dammam to Ras Tanura.

Production has been obtained in Kuwait, and tests are being made at Abu Hadriya, midway between Kuwait and Dammam.

Bahrein has a reserve of 150,000,000 brl. of oil, and Dammam is expected to have 300,000,000 brl. There are other geophysical highs.

G. D. H.

224.* **General Considerations in Estimating Reserves of Petroleum Capable of being Produced at the Different Concessions (Venezuela).** M. Guadalajara. *G. Rev. del Min. Fomento (Venezuela)*, September 1939, II (16), 491-510.—The problem of estimating the reserves of petroleum economically obtainable from an oil-field presents obvious difficulties. The author discusses various methods and presents calculations and curves. A commonly used method is to estimate the volume, porosity, and saturation of the sand or rock containing the oil. For example, if the area in question is 20 million sq. ft., the sand 25 ft. thick, spacing of the wells $\frac{1}{6}$ of the area, saturation degree $\frac{1}{2}$, recoverable oil $\frac{1}{2}$, then the total production would be $20,000,000 \times 25 \times \frac{1}{6} \times \frac{1}{2} \times \frac{1}{2}$ cub. ft. of oil. When records of the outputs of wells in a field are available, curves may be plotted for the areas and districts. Such curves will generally show a certain percentage reduction from year to year, and in this way it is possible to estimate future production. Particulars are given of other methods in use.

Attention is directed to various possible sources of error, such as the conditions arising when all the wells in the field are not sunk at the same time, in which case the later wells will affect the results, due to interference. Similarly the abandonment of some wells will increase the output of the remainder. Other variants are fully discussed.

Probable outputs are worked out for a number of fields based on the curves shown, but it is pointed out that not until 1930 were continuous records regularly kept. In these calculations the curves of declining output are extrapolated to the years when further extraction would not be considered economical.

The author has gone to a great deal of trouble in looking up such records as are available in order to arrive at the conclusions in respect to future outputs given in this article.

He winds up by saying that it will be seen that the work can only be regarded as a trial effort which may serve as an introduction to future studies which may be undertaken in his country.

H. I. L.

Geophysics.

225.* **Refraction and Reflection of Seismic Waves. II. Discussion of the Physics of Refraction Prospecting.** C. H. Dix. *Geophys.*, 1939, 4 (4), 238-241.—The physical process by which the refracted wave is generated is discussed. After the pulse from a given source reaches the critical position where the refracted wave-front is normal to the interface, the wave travelling in the lower medium starts to draw away from the pulse in the upper medium. As a result of stress relationships and the conditions of continuity at the interface, the pulse in the lower medium must give rise to a disturbance in the upper, and this travels along the interface with the velocity of waves in the lower medium. It is this travelling disturbance which is the source of the wave used in refraction prospecting. The intensity depends on the elastic constants and densities of the two media, the distance of the source from the interface, the variation of velocities in the lower medium, the curvature of the interface, and the intensity at the shot point. The effect of velocity variation and curvature of the interface is briefly discussed.

The effects of shear-waves are not mentioned, but as the process applies equally to them, it may be expected that each travelling source of disturbance will generate both shear and compression waves.
S. E. C.

226.* **Seismic Paths, Assuming a Parabolic Increase of Velocity with Depth.** C. E. Houston. *Geophys.*, 1939, 4 (4), 242-246.—In the case where velocity is a function of depth only, $v = v(y)$, various forms of the function $v(y)$ have been suggested. The assumption of a linear variation of velocity with time leads to a parabolic velocity depth function, and this example is discussed in the present paper. It is of interest in that the equation of the wave-paths can be obtained, the time-distance curve of the direct wave can be computed, and the corresponding reflection time-distance curves can be obtained graphically.
S. E. C.

227.* **Areal Plan of Mapping Subsurface Structure by Refraction Shooting.** L. W. Gardner. *Geophys.*, 1939, 4 (4), 247-259.—The following methods may be applied to areas in which the beds are more or less level, and in which there is at least one suitable marker horizon. In new areas it is necessary to carry out preliminary shooting to determine the necessary working conditions. With the detectors located at suitable distances from shot points, the sum of the two delay times at positions offset from the shot point and detector positions for each set-up may be determined. The average of the delay times may be generally regarded as giving a rough measure of each, but methods are described whereby the relative and absolute delay times are obtained from geometrically interrelated set-ups. These methods are applicable when approximate values of offset distances are known. If shots are made on two set-ups arranged to have a common offset position, the difference in depth at the two free ends may be calculated. The system can be extended to a group of set-ups with the detectors arranged on a ring around the common offset. Undulations of the marker horizon below this ring will then be determined. Another arrangement involves three set-ups arranged in triangular form, such that each offset position of one line coincides with one offset position of an adjacent line. By this means the absolute delay times, and hence depths, may be determined. By combining these two methods in the form of a system of triangles and hexagons, it is possible to shoot over an area and determine the depths and undulations of the marker horizon.
S. E. C.

228.* **Dip Reflections on Two Faults in the Gulf Coast.** F. F. Campbell. *Geophys.*, 1939, 4 (4), 260-270.—The algebraic sum of the dip determinations around any particular traverse is termed "misclosure," and the problem then is to decide whether this misclosure is due to errors in observation or to faulting. The results of two surveys in the Gulf Coast are given in illustration of the problem. In one case the amount of misclosure and the quality of the work make it apparent that faulting must be present. In the other case geological evidence was necessary before the presence of a fault could be assumed. No sharp line can be drawn between the effects of faulting and those of observational errors. It is recommended, however, in the case of a large misclosure on

a reconnaissance survey shot with less than 100% control, that the original traverse be cut up with lines shot for 100% control. Then, if the misclosure is concentrated by the additional shooting, faulting is the probable explanation. S. E. C.

229.* Shallow Resistivity Survey at South Elton, Louisiana. E. E. Blondeau. *Geophys.*, 1939, 4 (4), 271-278.—The survey was made in the usual manner with standard D.C. resistivity equipment. Conductivity contours were drawn for the cases where the potential electrodes were 100 and 300 ft., 400 and 600 ft., and 900 and 1100 ft. from the near current electrode. The contour map on the deep zone is comparatively uniform, whilst that on the middle zone shows a little more variation. The conductivity contours on the shallow zone, however, are extremely complex. The results obtained on the shallow zone are compared with an Eltran survey in the same area, and excellent agreement is obtained.

In discussing the cause of the wide conductivity variations in the shallow zone, it is pointed out that the anomalous portions of the section are high conductivity strips of surface material. If the cause of the high conductivity is mineralization, then it must be purely a surface phenomenon. The more probable cause, however, lies in the history of the surface-beds which are of deltaic and lagunal origin. Any association of these shallow anomalies with deep structure is highly improbable. S. E. C.

230.* Note on the Relation of Suddenly Applied D.C. Earth Transients to Pulse Response Transients. G. E. White. *Geophys.*, 1939, 4 (4), 279-282.—The experimental difficulties of obtaining the derivatives of the voltage transient from the suddenly applied D.C. results are practically insurmountable. If, however, a sharp D.C. pulse is applied to the earth, lasting, for instance, less than 0.001 sec., then the observed transient would approximate to the time derivative of the transient obtained from the suddenly applied D.C. case. It is shown that a definite possibility exists of measuring this type of response, using reasonable power voltages. S. E. C.

231.* Recent Developments in Eltran Prospecting. P. W. Klipsch. *Geophys.*, 1939, 4 (4), 283-291.—Difficulties encountered in the null method have been overcome by the introduction of a new mixing circuit, a new means of synchronizing the opposing wave with the detected wave, and a simplified synthesizing circuit. These are described, and it is shown that by combining the synthesizing circuit with a low-power, square-wave generator separate from the rest of the circuit, it is possible to calibrate the rest of the apparatus.

A new method of measuring the wave-shape distortion—the Sawtran method—has been introduced. Instead of producing a wave form which opposes the detected wave and produces a null, the detected wave is subjected to further distortion until a pre-determined wave-shape is produced. The method measures a single characteristic of the detected wave, such as the time constant, and its use is a distinct advantage in areas where the wave-shape is not too complex.

The use of cameras in conjunction with the cathode-ray oscillograph is an advantage, in that they afford permanent records, they give checks on data collected by dial readings, the wave-form can be detected through an electrical haze, and the records can be subjected to detailed analysis. S. E. C.

232.* Proposed Geophysical Method for Orientating Cores. V. Vacquier. *Geophys.*, 1939, 4 (4), 292-299.—It is suggested that a short pilot-hole be drilled and this hole then filled with a suspension of highly magnetized particles, the suspending medium being such that it will solidify and adhere firmly to the rock. The upper part of a core subsequently taken by normal coring methods will then contain the polarized, magnetic material and can be tested with a portable magnetometer. In this manner the core may be orientated. S. E. C.

Drilling.

233.* Test Indicates Potentialities of Diesel-Electric Rig for Deep Drilling. H. F. Simons. *Oil Gas J.*, 30.11.39, 38 (29), 32.—This long paper is the first of a number appearing in a special section of the November issue of the *Oil and Gas Journal*, and describes

Mid-Continents deepest test—Continental Oil Companies' No. 1 Proctor, which was recently plugged back to 14,582 ft. The chief object of the test was to discover geological facts which could indicate the trend of further prospecting in the area.

More than 15 months were required to drill the test to its total depth, and the last 3 months were spent mostly in fishing. A full description of this phase of the work is given at the end of the paper. At present the test is active, plugging back and testing operations being in progress.

The drilling rig used was a diesel-electric rig, which has been used both in other deep wildcats and in proven fields. The main units were two Fairbanks-Morse, opposed-piston, two-cycle, six-cylinder, 300-h.p. diesel engines with a speed range from 900 to 1200 r.p.m. Each engine drove directly a 140-kw. and a 50-kw. direct-current generator and a 25-kw. exciter through V-belts. Details of engines, generators and exciters, and of the panelling arrangement are given.

The arrangement gave great flexibility, as three circuits were available to the driller. Any one generator or any combination of generators could be connected to any motor desired. Examples are given of different systems of connections for various drilling, hoisting, and mud-mixing operations.

The description of the switchboard reveals many interesting safety arrangements which render it foolproof.

Many details are given of the actual equipment used in drilling this test well, including derrick equipment and drill-pipe used. Every precaution has been taken by the operators to prevent damage to the equipment, possible blow-outs, and injury to men. The preventer used may be operated by mud, water, or air pressure, with valves for its control located on the floor. Safety precautions are summarized.

The paper then describes the operations undertaken in, and the history of, drilling the test. The drill-pipe stuck at a depth of 14,479 ft., and many attempts were necessary before it could be freed. A description of this fishing job has already appeared in the Press (*Oil Gas J.*, 28.9.39). The fish was finally side-tracked.

A. H. N.

234.* Rôle of Metals in Deep-Well Drilling. W. L. Nelson. *Oil Gas J.*, 30.11.39, 38 (29), 37.—The difficulties which are encountered in deep-well drilling and the severe conditions existing may be emphasized by noting a few facts about operations similar to those of Proctor No. 1, such as:—

- (1) At 14,479 ft. the stretch of the drill-pipe is 12–14 ft., or about 0.1%. Many ordinary carbon steels would be permanently deformed under such a load.
- (2) Derricks are required to support more than 500,000 lb., or 250 tons—equivalent to a bundle of 167 automobiles, each weighing 1½ tons.
- (3) Bits having a cutting diameter of only 4 or 5 in. must sustain a load of 1000–1500 lb./sq. in. of hole. If bits were rods, this stress would not be excessive; but they are movable mechanisms, and much of the load is centred on the cutting edges rather than evenly distributed, and, furthermore, the bits revolve two to four times per second.

The development of new steels is described, and it is seen that such developments are gradual processes. After laboratory studies, a proposed steel is put into commercial operation and carefully watched for months or years. Should difficulties develop, they are overcome by heat-treating the new batches of steel in different ways. These experiments take years, and hence steel practice must be summarized over a period of years rather than day by day. Such a summary is made.

The paper gives in tabular form strength and hardness of oil-field steels and their approximate relative cost, and examples of modern and older steels used in drilling operations.

From the text and the tables it may be summarized that, from the standpoint of strength alone, present-day steels range from 50% to even 250% stronger than those used in early drilling practice. The values of greater hardness, wear resistance, freedom from failures, and increased speeds of drilling are factors which make it impossible to evaluate the real advantages of the newer steels.

A. H. N.

235.* Mud Control is Big Problem on Continental Deep Test. D. B. Edwards. *Oil Gas J.*, 30.11.39, 38 (29), 38.—Practices employed in the drilling of Proctor No. 1 were not

commonly used in Mid-Continent drilling, and hence mud treatment was unusual. Only one mud change was necessary—between 10,176 and 14,491 ft. Chemical treatment and the use of artificial mud made it possible to maintain a mixture which had low water loss, low initial gel strength, moderate 10-minute gel strength, and thin cake-forming qualities, despite the contamination from salt-bearing formations, acid and oil used in attempting to free stuck pipe, and the drilling of cement.

The conditions existing in the well at various times determined the type of mud necessary and the treatment which was needed to obtain it. A brief description of the drilling of the well and equipment used is therefore given, to establish the background for the mud treatment.

The artificial mud was used until a depth of 3500 ft. was reached, when aquagel was added to bring the viscosity to 35 sec. Below 5000 ft. the viscosity was kept between 35 and 40 sec. A.P.T., and the density ranged from 9.8 to 10.2 lb./gal. Loss of mud became troublesome below 9250 ft. This was undoubtedly due to the flocculated condition of the mud, resulting from its high salt content. Because of losing mud into the formation, a good sealing mud was needed which would release gas easily at the surface; yet it was essential that the mud should have sufficient viscosity to carry out the cuttings and sufficient gel strength to hold the cuttings in suspension when circulation ceased. A table is given showing the limits set on the properties of the mud which possessed all these desirable qualities.

The control necessary to keep the mud within these limits is detailed. A derrick man tested the weight and Marsh funnel viscosity of the mud at the well at regular intervals of 15–30 mins., and regulated the flow of the chemical solution being used to treat the mud at the time in accordance with the instructions received from the engineer or the tool-pusher.

Other tests on the mud, such as the Stormer viscosity, gel strengths, pH value, and filtration qualities, were made daily by the engineer. These tests indicated the type and quantity of admixtures to be used in the treatment of the mud.

The mud used in drilling the lower part of the well had four peculiarities: (1) a very low water loss; (2) a thin cake formation; (3) extremely low initial gel strength; (4) moderate 10-minute gel. These factors and the various controls used are described in detail, and the advantages and drawbacks of each are stated.

The effects of the acid and a study of pH values of the mud are of particular interest.

A. H. N.

236.* Metals in Drilling. W. L. Nelson. *Oil Gas J.*, 30.11.39, 38 (29), 69.—This is the ninth of a series of papers devoted to the metallic materials used in drilling wells. Previous articles have dealt with portable servicing equipment, slush-lamps, rotary-chain, rotary-table, hoists, engine-castings, pulley-blocks, and bits.

In this paper the metals used in tubular drilling equipment are discussed. Inspection of equipment, troubles and difficulties arising from manufacturing points of view—such as the large volume of the equipment preventing easy heat-treatment—or from the view-point of use, such as the severe and complex stresses arising in a drill-pipe subjected to twisting, bending, tension, and even compression stresses, are all briefly summarized. A table is given showing the approximate composition, in terms of per cent. carbon, manganese, chromium, nickel, and molybdenum, the approximate physical properties in terms of yield point, elongation, and Brinell hardness, and the heat treatment of metals used in tubular drilling equipment. The properties discussed have been taken from published statements of metal manufacturers, fabricators, and field-men. References are given.

A. H. N.

237.* Safety When Drilling Deep Tests. Anon. *Oil Gas J.*, 30.11.39, 38 (29), 76.—In this short article are summarized various precautionary arrangements found to yield an increased factor of safety in deep drilling, particularly in unproven localities. Actually it appears that men on deep tests are generally in less danger of injury, despite the increase in the work, than those on shallow tests, because of safety appliances and practices in use.

A photograph of an actual "safe" rig is reproduced and the various items are described, together with their advantages.

A. H. N.

238.* Shell Overcomes Caving to Bring in Coastal Well. N. Williams. *Oil Gas J.*, 7.12.39, 38 (30), 55.—Excessive cavity conditions and caving formations encountered by a certain company in drilling one of its wells in coastal Louisiana presented many difficulties and problems that would ordinarily have discouraged most companies from continuing operations. However, through persistent efforts, involving a number of unusual procedures, these were successfully overcome, and operators were finally able to complete the well as a good producer.

The paper describes in detail the many and varied operations involved in drilling and completing this unusually difficult well to a total depth of 7704 ft. In addition to the numerous cement plugs used and conventional muds and chemicals provided in attempts to plug and seal cavities in porous formations, a total of 1475 bales of Fibrotox, 200 sacks of cotton-seed hulls, 80 sacks of rice bran, 21 tons of sawdust, and 5444 empty sacks, including gunny-sacks, mica, and cloth-cement sacks, were pumped into the hole. Altogether 124 days were required in overall time for drilling the well from the time the hole was spudded until its completion. A. H. N.

239.* Metals in Drilling. W. L. Nolson. *Oil Gas J.*, 14.12.39, 38 (31), 53.—The paper deals with miscellaneous alloy equipment. These pieces of drilling equipment are of such a varied nature that they cannot be treated as a whole. Because of the extreme importance of cost, a brief and general comparison of the relative costs of the various low-alloy steels is included. The alloys that are recommended have been reviewed in published information from recognized steel companies, fabricators, and from discussions with field men.

The approximate compositions, in terms of percentage carbon, chromium, nickel, and molybdenum, and the approximate physical properties (yield point, elongation, and Brinell hardness), together with suggested heat treatments, are given for such pieces of drilling equipment as tool-dressing implements, tong handle, inserts, link-pins, kelleys, drill-jars, etc. A. H. N.

240.* Oklahoma Deep Test Well. B. Mills. *Oil Wkly*, 27.11.39, 95 (12), 39.—In this lengthy paper the author describes drilling, fishing, and cementing operations undertaken in the second deepest well, Proctor No. 1, together with geological structures and other interesting items. Drilling to 14,582 ft. with medium-weight equipment points to future depth records far below those currently regarded as "deep."

The chief outstanding mechanical developments in drilling Proctor No. 1 were: cementing 9½-in. casing at 10,173 ft.; cutting out 2250 ft. of drill-pipe between 12,229 ft. and 14,464 ft., squeeze-cementing of porous formations below the 9½-in. casing, and successful use of diesel-electric equipment. Each of these achievements affords interesting study.

Mud conditioning and control are described and certain peculiarities of the mud noted, such as the low initial gel strength attained and the advantages accruing therefrom.

Diesel-electric power has been used exclusively for drilling this deep test. The equipment and power connections are detailed. Fuel consumption runs from 400 to 900 gals. daily.

A table gives the subdivisions of the formations which have been encountered. Several shows of gas have been noted between 10,700 ft. and 13,400 ft. in thin, sandy limestones and conglomerates, the porosity of which is relatively low (shown by electrical survey). It is planned to test thoroughly all shows before completion. A. H. N.

241*. Rotating while Cementing Proves Economical. B. Mills. *Oil Wkly*, 4.12.39, 95 (13), 14.—Constant rotation of casing during cementing operations has become an important phase of completion operations in one field in South Texas. This novel practice has been used by one company while cementing the oil string in about 175 wells, and no failure has been recorded.

A sectional view, as well as a photograph and description of the equipment used, are given in the paper. The basis for successful rotation of casing during cementing operations has been straight holes, long threads and couplings or casings, assurance

that casing is free before rotation starts, and suitable rotating equipment. Rotation has been applied only to cementing full oil-strings.

Completion depth in this particular field averages 5925 ft. The 5-in. casing-string is rotated steadily for an hour in an 8½-in. hole, the rotating time including the mud-conditioning, cement-mixing, and pumping-down periods. The casing is rotated from 25 to 30 r.p.m., and rotation stops about the time the cementing plug reaches bottom. Power required to turn the casing is negligible. No damage to clamps or dies used to rotate the casing has occurred.

A. H. N.

242.* Quick Means for Finding Number of Barrels in a Casing String. W. F. Schaphorst. *Oil Wkly*, 4.12.39, 95 (13), 30.—This is a nomograph giving the capacity in barrels of strings of casing, given the diameter and depth of the well. The way of using the simple chart is illustrated.

A. H. N.

243.* Michigan Gets its First Natural Flowing Trenton Lime Well. N. X. Lyons. *Oil Wkly*, 11.12.39, 96 (1), 36.—The first high-potential, naturally flowing Trenton limestone well, source-bed of Ohio's oldest oil-fields, ever developed in Michigan, is causing considerable interest, but is not likely to precipitate anything in the proportion of a boom.

The drilling practice and completion methods followed in bringing in this well are detailed, as well as the financial and other interests; and the geological structure and previous production records of the field are discussed.

A. H. N.

244.* Temperature Effects on Oil-Well Drilling. Part I. M. T. Halbouty. *Oil Wkly*, 18.12.39, 96 (2), 10-16. Paper presented before American Institute of Physics.—As wells have been drilled to constantly greater depths, increasingly higher temperatures have been encountered, and it is now recognized that this condition will have important influences on oil and gas drilling and producing methods. Consequently this paper on the subject is of interest and value. No attempt is made to review every particular phase where temperature affects drilling and production, only those cases where temperatures have an influence from an economical standpoint and how these effects are dealt with scientifically being discussed.

Because of its length, the report is divided into two parts for publication. Part 1 discusses temperatures affecting oil-well drilling; Part 2 describes temperatures affecting crude-oil production, and discusses the geothermal gradient present in Gulf Coast oil-fields.

One of the most important effects of temperature on drilling is that of increased temperature on drilling mud. Whilst the effects of higher temperature on the density of muds are negligible, they add to the problems of mud control by increasing the mud's viscosity—hence making viscosity control more difficult—and also by decreasing the effectiveness of the mud in its wall-building operations in the hole.

It is believed that, despite the cooling effects of the drilling mud on the bit, high-friction temperature is perhaps the principal reason for a case-hardened rock-bit drilling less than 10 ft. of hole in extreme cases.

It is only recently that high bottom-hole temperatures have seriously impaired cementing operations. When drilling depths increased to below 6000 ft., the bottom-hole temperatures of 150-170° F. that were encountered rapidly increased the setting time of ordinary cement. This fact, together with the additional delay in pumping cement through 6000 or 7000 ft. of casing, has been directly responsible in several cases for the cement setting inside the casing and becoming immovable when a small part had passed out through the casing shoe, thereby improperly cementing the hole and causing unnecessary delay in drilling out several hundred feet of cement in the casing. These facts and their effects on the development of improved cement are discussed. Methods for the location of cement behind casing by temperature measurements are illustrated.

This part of the paper ends with a discussion on electrical temperature logging with survey instruments, correlation in drill-holes by means of subsurface temperatures, location of gas-oil levels and points for the entry of water into the well, and finally with a discussion on the economic importance of temperature measurements.

A. H. N.

245.* Drilling Activity Continues above 1938 Trend. L. J. Logan. *Oil Wkly*, 18.12.39, 96 (2), 36-38.—Drilling activity in the U.S.A. during the first eleven months of 1939 is detailed in pictorial and tabular form. November activity brings the number of well completions for 11 months of 1939 near the 1938 level, whilst the field activity on December 1st was considerably greater than that existing in the corresponding period of 1938.

A. H. N.

246.* Simultaneous and Continuous Electric Logging and Drilling Achieved. B. Mills. *Oil Wkly*, 1.1.40, 96 (4), 16-20.—Continuous electric logging of formations while simultaneously conducting regular oil-field drilling operations is described, and the various implements for carrying the circuit to the bottom of the hole are shown pictorially.

The circuit consists principally of a battery connected to a meter, which in turn is connected to leads running to the derrick. The main lead runs up the standpipe, around the rotary hose, to a brush-box containing an ordinary collector ring and a set of brushes. The circuit is then taken past the kelley by a special conductor. Thence it leads down the drill-pipe through special connections to the drill-collar assembly, which consists of an insulated bakelite section above the bit, a testing-switch and a special steel drill-collar to provide weight and rigidity.

Although only one cable is used between the bit and the surface, both the earth potential and the impedance are recorded on meters at the surface. A small alternating-current oscillator is used to energize the cable. The impedance meter is not affected by the direct current, whilst the potential or direct current is not affected by the impedance measurement. The earth potential is measured in millivolts, the resistance in ohms. The earth potential is measured direct from the bit as direct current.

The log made while drilling is checked by re-surveying the hole. Part of the drill-pipe is pulled, a dummy is snapped on, and the bit run back into the hole as a check. It is also easy to go back into the well and pick up portions of the hole fully surveyed while drilling.

The drill-pipe and most of the other equipment are handled as if they were conventional items. The pipe itself is standard grade, only the pressed-in connections in the tool-joints and the brazed conduits running down each joint differentiating the assembly from an ordinary drill-pipe. Experienced operators are, however, kept on the well, as interpretation of the electric log involves technicalities not usually understood by an ordinary drilling crew.

A. H. N.

247.* Heaving Shale Research Programme is Instituted. Anon. *Oil Wkly*, 1.1.40, 96 (4), 32-33.—The paper outlines the future programme of research to be carried out in the University of Texas, but the analysis of the problem in designing the programme renders it of general interest. It appears that the problem may be classified under several distinct divisions, the major ones being (1) regional studies; (2) types of heaving shales; (3) causes of heaving shales; and (4) combating heaving shales.

An outline of the basic principles and the programme of research to solve the problem is given.

A. H. N.

248.* Drilling and Completion of 10,000-ft. Well in Louisiana Gulf Coast. H. L. Flood. *Petrol Engr*, December 1939, 11 (3), 29.—The most important discovery of the year on the Gulf Coast of Louisiana is a very recently completed well. This is the second well to be drilled on the structure, located apparently on the flank of the structure, drilled through a fairly thick section of gas and condensate formation, but drilled deeper to discover one of the thickest, and what may be one of the most prolific oil reservoirs discovered recently in Southern Louisiana. A total of 140 ft. of oil-saturated sand was logged in the well, of which only 45 ft. is being produced through perforations in the casing. Potential of the well is 1710 brls./day through $\frac{3}{8}$ -in. choke. Pressure on the tubing-head is 1750 lb./sq. in.

The drilling history of the well and the equipment used are given. Drilling of this well has been characterized by lack of trouble—a fact attributed not only to the experienced crew, but also to the accurate mud control undertaken by a competent mud engineer on the job. This practice of relegating mud control to one responsible expert is advocated for the success of any deep-drilling project.

A. H. N.

249.* Deep Test Drilled by Diesel-Electric Rig H. L. Flood. *Petrol. Engr*, December 1939, 11 (3), 42.—An extensive account is given of the drilling history of, and equipment used for, the second deepest hole in the world. The most interesting features of the equipment used were:—

- (1) The installation of a complete auxiliary hoisting unit and pump as a standby;
- (2) The re-designed electrical controls permitting extreme flexibility in operation;
- (3) The opposed-piston diesel engines used to drive the generators.

The opposed-piston diesel engines employed on this rig have not been used extensively as drilling power, although the design is widely employed in marine service. Operating on the two-cycle principle, these six-cylinder engines have, in reality, twelve cylinders. Six pistons, each 5 in. in diameter, and having a 7-in. stroke, are attached to a crankshaft above, and six pistons are attached to a crankshaft below. Instead of compressing against a fixed head, as in an ordinary engine, the pistons compress against each other. The expanding gases resulting from the explosions thus exert force against both pistons simultaneously. The lower crankshaft is directly connected to the generators; the upper crankshaft is linked to the lower one by an enclosed chain drive. A detailed description of this engine appeared in the *Petrol. Engr*, May 1936. A. H. N.

250.* New Pocket Transit Combines Functions of Several Instruments. L. Suverkrop. *Petrol. Engr*, December 1939, 11 (3), 69.—A description is given of a light-weight instrument designed for the use of engineers and geologists doing reconnaissance work which serves as compass, transit, level, clinometer, sextant, and alidade. Detailed account is given of the methods employed in each type of work. A. H. N.

251.* Characteristics and Requirements of Portable Well-Servicing Units. W. K. Acheson. *Petrol. Engr*, December 1939, 11 (3), 121.—*Paper presented before Petroleum Transport Association.*—Performance of well-servicing units differs from that of most other types of equipment that operate from an engine. In pulling rods or tubing it is necessary to start and to accelerate the load without changing gears until a stop is made to unjoint the rods or tubing—a condition imposed because there is no momentum built up to carry the load while the gears are shifted.

Fundamentally, all well-servicing units are similar. The ability of any servicing unit to move and to accelerate a given weight depends on the power characteristics of the engine and the speed reduction provided in the gears of the transmission, chain sprockets, and the number of lines strung through the blocks.

The author discusses the characteristics of engines and gives the method of determining these characteristics. The change of torque with speed and the brake h.p. developed at various speeds are studied from first principles. The pulling ability of the engine is analysed, and the effects of clutch characteristics, gear ratios, and number of lines in the blocks are illustrated by simple examples which are completely worked out. A. H. N.

252.* Radial Filtration of Drilling Muds. M. Williams. *Petrol. Tech.*, November 1939, A.I.M.M.E., Tech. Pub., No. 1112.—The filtrate which penetrates the strata may present a serious problem in many instances. The factors which determine the rate of such infiltration have been studied, and a series of curves and equations has been developed which permits the distance of penetration to be estimated. Since the dimensions of the hole and the rate of mud circulation are usually fixed by drilling conditions, variation of these to reduce filtration is impracticable. Mud control is the obvious solution.

Each mud presents a different problem with regard to securing optimum filtration properties, but there are certain general facts: (1) dispersing agents reduce filtration, while coagulating agents increase it; (2) the predominance of particles of colloidal dimensions ordinarily is conducive to low filtration rates.

Experimental and theoretical treatment of axial and no-flow conditions is given, and the possibility of filling the hole with a filter-cake is considered unlikely to occur except with a mud of exceptionally bad filtration characteristics. G. D. H.

253.* **Detection of Radioactive Cement in Cased Wells.** L. G. Howell and A. Frosch. *Petrol. Tech.*, November 1939, A.I.M.M.E., Tech. Pub., No. 1113.—An early apparatus has been modified so as to permit measurement of the relative intensities of gamma rays from radioactive elements occurring naturally in geological formations along the walls of a bore-hole and behind the casing. Comparison of the absolute magnitude of the open-hole and cased-hole logs reveals that there is some absorption by the casing, but the outstanding features of the log are still maintained, and the gamma-ray log shows a remarkable correlation with electrical logs. In general the sands show up as radioactive "lows" and the shales as radioactive "highs."

If radioactive material, such as carnotite, is added to cement, the apparatus can be used to detect the position of the cement. The method is more flexible than the temperature method, in that it is not necessary to make the survey while the cement is still setting; the circulation of the mud has no disturbing effect; the concentration of ore added to the cement can be varied to give a sufficiently large change in gamma-ray intensity even though the treated cement may be pumped behind old cement. However, it must be remembered that the location of formations behind radioactive cement cannot be made with this method since the feeble radioactivity of the formations is masked by the strong radioactivity of the cement. G. D. H.

254.* **Effect of Temperature on Plastering Properties and Viscosity of Rotary Drilling Muds.** H. T. Byck. *Petrol. Tech.*, November 1939, A.I.M.M.E., Tech. Pub., No. 1116.—The plastering properties of six representative Californian drilling muds have been studied over a temperature range of 70–175° F. at several mud weights, using a high-pressure circulating filter press with full-size consolidated cores. In all tests, with untreated as well as with chemically treated muds, the filtration rates at elevated temperatures were higher than at low temperatures, and in nearly every case this increased filtration rate was notably larger than could be attributed solely to the decreased viscosity of water at elevated temperatures. It appears that no existing method will permit even an approximate determination of the filtration rate at high temperature from data at room temperature. It is necessary to make measurements at the temperature expected in the well, or to make a sufficient number of tests at lower temperatures so that a small extrapolation of the data to the expected well temperature may be applied. Such tests should be made on the mud as used in the well.

The viscosity of each of the muds was determined over the same temperature range. While no relationship was noted between mud viscosity and plastering properties, it was found that chemical treatment of the muds for viscosity reduction shifted the temperature of minimum viscosity to appreciably higher values than for untreated muds. G. D. H.

255.* **Study of Some Phases of Chemical Control in Clay Suspensions.** A. D. Garrison and K. C. T. Brink. *Petrol. Tech.*, November 1939, A.I.M.M.E. Tech. Pub., No. 1124.—The viscometer used was of the rotating cylinder type with an inverted hollow cylinder as rotor in a mud-filled annulus. The speed was capable of variation and the torque could be observed. The data are given for approximately equilibrium conditions. Measurements on kaolin, silica, sulphur, and barium sulphate suspensions gave straight lines when the viscosity (ordinates) was plotted against the reciprocal of the rate of revolution, and their slopes and the intercepts on the viscosity axis are in the order of the concentration. It appears to be a general principle that the intercept is higher the greater the relative volume of the suspended solid, and the slope is higher the greater the tendency for the solid particles to adhere together. It appears also that the more rapid the rate of flow the smaller becomes that part of the viscosity that is due to the inter-particle forces of attraction.

The viscosity-speed relations for bentonite suspensions are substantially different from those for relatively non-colloidal solids. The curvature obtained was demonstrably due to the presence of very small particles.

The viscosity of a clay suspension is due to: (1) the viscosity of the suspending medium; (2) the viscosity normally attributable to the obstructive effects of solid particles; (3) the forces between the solid particles tending to give relative rigidity; (4) hydration of the particles. The curvature shown by bentonite suspensions at high rates of revolution is attributed to loss of the hydrated envelope on the particles.

The effects of adding various chemicals to the suspensions have been studied and interpreted in the light of these possible viscosity factors. The deflocculating agents that have commonly been used seem to be those which influence structural viscosity only. To control hydration is not so simple. It involves such methods as an exchange of ions on the clay surface for less hydrated ions, or a complete removal of such ions as is obtained during electro dialysis, or by the dehydrating effect of an excess of some water-soluble salt or of dehydrating liquids such as alcohol or acetone.

G. D. H.

256.* Dispersion of Clays and Shales by Fluid Motion. A. D. Garrison, K. C. T. Brink, and P. B. Elkin. *Petrol. Tech.*, November 1939, A.I.M.M.E. Tech. Pub., No. 1125.—It has long been recognized that the viscosity of a clay suspension depends on the past history of the fluid motion. In examining the effects of long-continued uniform motion a concentric cylinder rotating viscometer was used, with a standard rate of rotation of 600 r.p.m. It was observed that in clay suspensions capable of forming rigid gel structures, the time required for the attainment of an equilibrium viscosity value was frequently as long as 30 min. On continued shear some suspensions showed an increase in viscosity after passing through a minimum. It was found that the increase in viscosity was dependent on the age of the suspension, its concentration, the pH value, the nature of the metallic ions associated with the clay, and the presence of certain deflocculating agents. It is clear that an increase in pH above 7, and up to 11.1 at least, involves a substantial increase in the rate at which the viscosity rises under continuous motion. This is probably associated with an increase in colloidal dispersion and the resultant increase in hydration.

On giving a twelve-hour rest to a suspension treated with alkali or alkaline-earth hydroxides there was a drop in viscosity and in pH value, presumably due to chemical changes. Examination of the distribution of particle sizes in stirred and unstirred clay suspensions showed an increase in the percentage of fine particles and/or of hydration in the former case.

The bearing of the work on drilling operations is discussed.

G. D. H.

257.* Drilling Patents. M. M. Kinley. U.S.P. 2,185,303, 2.1.40. Appl. 1.7.38. Wire-line shooting-tool comprising a body, with an opening for, and means for gripping the wire line and shearing it at a point below the gripping point. Both the gripping and shearing mechanisms are actuated positively.

W. P. Rogers. U.S.P. 2,185,316, 2.1.40. Appl. 13.8.38. Deep well-drilling apparatus with a mast for a derrick suitable for cable-tool drilling.

G. E. Green. U.S.P. 2,185,337, 2.1.40. Appl. 7.1.38. Machine for slitting well-casings and the like.

S. P. Tschappat. U.S.P. 2,185,478 and 2,185,479, 2.1.40. Appl. 3.4.37 and 13.5.37, respectively. Casing- and tubing-heads.

N. C. Brewer. U.S.P. 2,185,702, 2.1.40. Appl. 8.7.38. Roller chain.

A. W. Buckner. U.S.P. 2,185,723, 2.1.40. Appl. 3.2.38. Cable clamp.

F. J. Young. U.S.P. 2,185,822, 2.1.40. Appl. 6.11.37. Rotary swivel.

A. H. N.

Production.

258.* Electrical Logs Locate Shots in Illinois Sand Wells. H. F. Simons. *Oil Gas J.*, 30.11.39, 38 (29), 59.—In efforts to improve the wells by shooting to maximum advantage, while at the same time minimizing the dangers of water encroachment, one company has introduced "shooting control" in the Loudon field in Fayette County, Illinois.

Under this method no well is shot until the electrical logs have been carefully studied by the geological department, which recommends the size of the shot and its location

in relation to the sand. Through this practice the amount of water produced has been materially reduced, and the wells are generally in good position to plug off water in case it becomes excessive.

The three productive sands of the Loudon field are described, together with the details followed before placing a shot. The effectiveness of the selective shooting-control method is evidenced by the large number of wells not so treated having a high percentage of water, with a consequent treating problem, whilst the wells carefully shot still have low water production.

A. H. N.

259.* Engineers Study Full Effect of Water Flooding in Bradford. J. P. O'Donnell. *Oil Gas J.*, 7.12.39, 38 (30), 43.—A study of a property that has almost reached the limit of its economic life as defined by present methods of production makes it evident that the Bradford field will still represent an oil reservoir of considerable magnitude after water-flooding has passed. This property will have a residual oil content of 10,238 brl./acre, or 56.2% of the original. However, the residual saturation will be but 25%. The field is the world's outstanding example of stimulating oil production by water-flooding. In recent years it has been producing from 37,000 to 49,000 brls. daily, 95% of which is being obtained from water-flooded properties.

The early development of the field from 1871 onwards is briefly outlined, and its life since it last changed ownership (1929) is detailed.

Eleven times as much water was injected as there was oil recovered, and five times as much water was produced as oil. The volumetric efficiency of this operation, on the basis of barrels of water injected to barrels of oil produced, is 9%. With 98.5% of the anticipated ultimate recovery already obtained, total recovery is estimated at 43% of the original content. The five-spot pattern was followed.

Tables and graphs give the characteristics of the property. A relatively unimportant figure in the discussion of water-flood properties is the actual life. This is because of the fact that during the first 5 years of development from 80% to 90% of the recoverable oil is produced, and the remaining 10-20% may be produced over a period of from 5 to 10 years. The principal controlling factor in the ultimate life of such a property is the price of the crude, which, after the production has fallen to an average of a fraction of a barrel per well per day, will, if high, prolong the life and, if low, will shorten it.

It was concluded that (1) the price of crude oil during the "depression decade" relegated water-flooding operations in this field definitely to the marginal class; (2) water-flooding, while it made continued operation of this field possible, was an inefficient, though effective, method of operation; (3) because of this inefficiency, the field will still represent an oil reservoir of some magnitude after production by present methods of water-flooding has been exhausted.

A. H. N.

260.* Circulating Hot Oil in Wells. L. P. Stockman. *Oil Gas J.*, 21.12.39, 38 (32), 34-35.—In order to reduce paraffin troubles and also the viscosity of the crude produced from deep wells in the Montebello field of California, one company has devised a means for circulating a portion of the crude. During circulation the crude is heated in an exchanger through which hot engine-cooling water passes. The heated crude is forced down the production line through a small pipe suspended inside the producing string.

A diagrammatic sketch of the heat-exchange system and several practical hints to ensure the success of the project are given.

A. H. N.

261.* Survey of World Oil-Fields. Anon. *Oil Gas J.*, 28.12.39, 38 (33), 76.—A complete list is given of the production activities of the world oil-fields under the headings of the respective countries in which they are located. The data are for (1) number of producing wells; (2) daily average production in barrels; (3) number of drilling wells; (4) producing depths in feet; (5) gravity of the crude, A.P.I.; (6) base of the crude; (7) outlet market for the crude; and (8) age of the field in years.

A. H. N.

262.* Steady Advance in Production Methods. Anon. *Oil Gas J.*, 28.12.39, 38 (33), 190.—This paper is published in the "International Number" of the *Oil and Gas*,

in both English and Spanish. It is an extensive review of production problems and their solutions, illustrated generously by photographs and diagrams. Well-spacing and organized drilling are the first problems discussed, followed by practices employed in modern rotary drilling requiring large drilling equipment and using more weight on the bit. Straight-hole drilling technique is discussed in conjunction with drilling-time studies, and power considerations, both from the prime mover and transmission view-points, are made.

In cable-tool drilling the outstanding development has been the perfection during the past year of a dynamometer that makes it possible to select the most suitable stroke. Cable-drilling technique is studied critically.

Electrical logging and core-orientation methods are reviewed. Completion practices are studied, and gravel packing, the use of removable pipe-sections in casing both by drilling and by acid action, and of cement-lined liners, are explained.

The problems encountered in flowing wells are briefly discussed before pumping wells are dealt with, attention being given to agitators and semi-agitators.

The paper ends with a review of the practices followed in gas-injection reservoirs, pressure conservation and restoration schemes, and finally in water-drives.

A. H. N.

263.* Range of Application of Gas-Lift Methods. Part I. E. C. Babson. *Oil Wkly*, 27.11.39, 95 (12), 15.—*Paper presented before American Petroleum Institute.*—This article presents the results of an investigation of gas-lift methods conducted under the auspices of the Production Practice Committee of the American Petroleum Institute's Division of Production. Pressure held against the formation and gas requirements are evaluated for: (1) straight gas lift; (2) plunger lift; (3) intermitters; and (4) gas-displacement pump. Comparisons of various methods are given and some suggestions made for application of the results to actual production problems. Because of the great length of the study, it is published in several parts.

In this, the first part of the paper, the author discusses the scope of the investigation and deals with cost of compressing gas, straight gas lift, gas lift through 2½-in. tubing at various depths and in various fields of the United States.

The behaviour of a producing well is the result of two sets of components: (1) characteristics of the well; (2) characteristics of the lifting equipment. The well characteristics depend on the behaviour of the producing formation and on the manner in which the well has been drilled, cased, and completed. In this discussion the chief items studied are (1) reservoir or static pressure; (2) productivity index; (3) formation gas-oil ratio; (4) per cent. water; (5) depth; (6) size and condition of casing; and (7) size and condition of liner.

The most important characteristics of any gas-lift method, on the other hand, are (1) back pressure held against the formations; and (2) gas consumption. The effectiveness of the installation is a function of the back pressure held against the formation; whereas the lifting efficiency is determined largely by the gas consumption.

The paper is particularly rich in graphs illustrating the various points enumerated here.

The pressures at the bottom of the tubing have been estimated from the casing pressures by applying the following equation, which has been found to give satisfactory results in the Dominguez field:—

$$P_B = (P_c + 15)(1 + 0.022h) - 15$$

where P_B = pressure at the bottom of the tubing, lb./sq. in.

P_c = casing pressure, lb./sq. in.

h = tubing depth, thousands of ft.

For deep wells it is assumed that the pressure at the bottom of a 10,000-ft. well would be identical with the pressure at the bottom of two superimposed 5000-ft. wells. The method is discussed and illustrated.

It is believed that the limits of the error of the curves covering costs of straight gas-lift through 2½-in. tubing and in the range of production rates of 100-400 brls./day and depths of 4000-6000 ft. are less than $\pm 10\%$. The limits of the error, however, are variable.

264.* Nine Deep Pumping Wells Have One Rod Break in 2½ Years. F. B. Taylor. *Oil Wkly*, 4.12.39, 95 (13), 24.—This is the twenty-eighth article in the series dealing with oil-well pumping problems. It describes the system at one lease where allowables are made without undue expense for breakdowns or replacements. On a basis of volume brought to the surface, the one-rod breakdown represents a total of 827,000 brl. of fluid between the time the various wells went on the pump and 1st November, 1939.

The operation of the pneumatic lifts installed is described. The polish rod is actuated through attachment to a double piston arrangement. Compressed air or gas is used to drive the power end, which is within the lower cylinder, whilst the upper piston rides under a cushion of oil.

Precautionary measures and safety devices against sudden dangers are described, as well as the performance of the equipment. The lease is outstanding in efficiency. The per barrel lifting cost is exceedingly low. The record is due not only to the equipment used, but also to the efficient, clear, and safe way the equipment is operated.

A. H. N.

265.* Range and Application of Gas-Lift Methods—Part 2. E. C. Babson. *Oil Wkly*, 4.12.39, 95 (13), 34.—Paper presented before the American Petroleum Institute.—Part I of this paper is abstracted under No. 263, and there the scope of the investigation and its ultimate purpose have been described.

In this, the second part, investigations are made on the cost of gas-lift operations in a 3-in. tubing similar to those undertaken on 2½-in. tubing in the first part. A great number of curves are presented and full sets of data are discussed in detail. The methods used here are the same as in the previous work on 2½-in. tubing, but where differences arise these are given in full.

In the range of 300–800 brl./day, 75–150 lb./sq. in. tubing pressure, and 5000–8000 ft. depth, the performance curves for 3-in. tubing are probably as reliable as those for 2½-in. tubing. On the other hand, the limits of error at low tubing pressures, low rates of production, and shallow depths may not be as satisfactory.

Information on gas-lift through sizes of tubing other than 2½-in. and 3-in. is too meagre to permit the development of reliable relationships.

Similarly, data on gas-lift through the annular space between casing and tubing are far from satisfactory, in that they are either insufficient in quantity or are poorly distributed over the range of conditions. As a result, the operating characteristics of casing-flow gas-lift can be established only partly. In an attempt to evaluate the characteristics of flow between 6½-in. casing and 2½-in. tubing, approximately 100 cases were obtained from production records. Pressures were estimated for the bottom of the tubing by a suitable formula, and graphs of performance plotted.

The data were unsatisfactory and the results obtained were consequently inferior in definition and importance to those obtained with the 2½-in. and 3-in. tubing.

The results of an experiment on gas-lift between 6½-in. casing and 2½-in. tubing in a 4800-ft. well are given.

A. H. N.

266.* Dual Separator Ensures Dry Gas for Power. Anon. *Oil Wkly*, 11.12.39, 96 (1), 18.—In this, the twenty-ninth article of the pumping series of the *Oil Weekly*, a description is made of a field pumping installation, using gas drawn from the mains serving a lease, in which a dual separator installation at the engine-house divides the regulator load and ensures proper dryness of fuel furnished to the engine.

A. H. N.

267.* Range and Application of Gas-Lift Methods. Part 3. E. C. Babson. *Oil Wkly*, 11.12.39, 96 (1), 22.—This is the third part of the exhaustive paper read before the A.P.I. on this subject. Further results are reported for gas lift between 6½-in. casing and 2½-in. tubing, and for gas-lift operations between 8½-in. casing and 2½-in. tubing.

A discussion on straight gas lift follows. The effects of water appear to be complex. The pressure at the bottom of the tubing is usually, but not always, higher in a wet well than in a corresponding clean one. The gas consumption of a wet well is often higher than that of a corresponding dry well—the explanation being the need for excess gas to prevent segregation of the water.

A hypothesis is advanced to explain the fact that some wells require very small quantities of gas for lifting oil.

Methods for calculating the energy available in 1 lb. of flowing mixture and for calculating bottom-hole pressures from surface readings are given. Graphs illustrate efficiency and density of mixtures at the well-head pressures.

The general types of flow-valves are described and the method of operation is outlined. When compressor capacity is limited, wells equipped with flow-valves can be produced with the minimum quantity of gas necessary to maintain steady flow. Should a well die, it will usually be kicked over automatically within a few minutes. Under curtailment, the value of the flow-valves increases. The important part played by flow-valves in making intermittent gas-lift methods practicable is discussed and other advantages are outlined. The chief contributions of flow-valves appear to be convenience and flexibility.

The working of plunger-lift devices is next studied in detail and its advantages are discussed. Results are reported for 2½-in., 3-in., and 4-in. plunger lifts. In a discussion on plunger lift the pressure at the bottom of the tubing appears to be obtainable from the formula :

$$P_B = P_s + KQH$$

where P_B = pressure at bottom of tubing, lb./sq. in.

P_s = minimum tubing pressure, lb./sq. in.

Q = rate of production, brl./day.

H = depth, thousands of ft.

K = a constant, 0.190 for 2½-in. lift; 0.115 for 3-in. lift; and 0.064 for 4-in. lift.

Part 3 of this paper ends with a discussion on closed-system intermitters. The paper is rich in diagrams and illustrations—forty-seven figures appearing in the first three parts.

A. H. N.

268.* **Effect of Reducing Pump Submergence.** M. L. Mayfield. *Oil Wkly*, 18.12.39, 96 (2), 17; also cf. *Oil Gas J.*, 14.12.39, 38 (31), 44, and *ibid.*, 21.12.39, 38 (32), 41.—By making tests and studying the individual well's producing characteristics, such as permeability, bottom-hole pressure, and productivity index, the efficiency of reducing the pump submergence can be predicted before permanent equipment is installed. It is concluded from the data obtained from the tests that where the practice of reducing pump submergence is applicable, definite advantages and disadvantages may accrue to the operator.

The advantages are: (1) The initial pumping-equipment investment may be decreased by \$1850 per well as compared with past practice. (2) The well is not damaged by this practice, and it is possible that some retardation of water encroachment may occur. (3) Operation and maintenance expense will be decreased, due to the smaller equipment and shorter rod-string. (4) Horsepower required will be reduced by 46–55%, or a saving of 4–7 h.p. per installation. Peak well-loads will be decreased from 8800 lb. to 4200 lb. at twenty-one strokes/min., i.e., a reduction of 52% or more is obtainable. (5) The application will extend from the initial installation time until the well is either fully depleted or has nearly reached this point. The savings effected by deferring cash requirement will more than offset the infrequent additional costs incurred should a larger unit be necessary for depletion.

It may also be concluded, from data on five leases, that the rate of decline of the static fluid level is 50 ft./year.

The disadvantages are: (1) Unless proper and sufficient pump submergence is obtainable and attention is given to the pump, "gas locking" may occur, which not only decreases the pump volumetric efficiency, but also causes unpredictable stresses to be imposed on the surface equipment. (2) Unless strict supervision and proper design of the sub-surface equipment are made to prevent misapplication of the smaller-rated units, a financial loss due to equipment failure may result. (3) Misplaced working barrels might create excessive pulling expense, although this could be limited by multiple barrel installation. (4) It is essential to make a preliminary test and detailed study of the individual wells before a permanent setting is made, and this will cost approximately \$150 per well.

A. H. N.

269.* **Equitable Proration of Marginal Leases in East Texas.** I. I. Gardescu. *Oil Wkly*, 25.12.39, 96 (3), 12-14.—Last June, when a Federal District Court declared the method of prorating wells in East Texas field void, owing to failure to consider acreage and oil in place, the ownership of many millions of barrels of oil not yet produced from the pay horizon was placed at stake. In view of the enormous reserve still in the ground—from 1750 to 2750 million barrels—a nominal change in the method of apportioning proration allowances might shift the ownership of a great volume of oil.

The East Texas field is considered a water-driven field, and, as such, for every barrel of oil recovered from a down-dip marginal lease many other barrels of oil have migrated from the lease, up dip, to be produced by wells located in more favourable structural locations.

The author explains the mechanism of production from such fields and discusses various suggestions made to render the proration of marginal leases in such fields equitable. A. H. N.

270.* **Temperatures Affecting Crude Oil Production.** M. T. Halbouty. *Oil Wkly*, 25.12.39, 96 (3), 15-19.—*Paper presented before American Institute of Physics.*—It is pointed out that while, in recent years, numerous reports have been published on the conservation of natural reservoir energy in the form of pressure, the study of sub-surface temperature, which provides another source of natural reservoir energy, and the effects of these temperatures on the flow of oil and gas through the sand, has been sorely neglected. The scope of the paper is limited to effects of temperature on production at the surface.

Solar heat is the principal factor to be considered in a discussion of the effects of temperature on production. A study is made of wax and paraffin deposits affected by temperature and methods for removing them or preventing their depositions. Chemical type of treatment may be either the type which generates heat, thereby raising the temperature of the oil and melting the paraffin or increasing the solubility in the oil, or the type which acts as a solvent to change the character of the wax so that it will not crystallize when the temperature is decreased below its original melting point. The practical difficulties encountered in applying either method are discussed. The use of steam, either in coils or by injecting it directly into the pipe, is next studied.

The economic importance of high bottom-hole temperatures is illustrated by reference to practices followed by various companies and the advantages accruing from such practices. Finally heat-treating crude-oil emulsions is made the subject of discussion on the effects of temperature on the economics of production.

The paper ends with a discussion on the geothermal gradient present in the Gulf Coast oil-fields. The apparent concentration of heat near salt domes may be partly explained by the fact that salt is an excellent conductor of heat, and assuming that the salt plug extends to depths in excess of 20,000 ft., the high temperatures existing at this depth would be more readily transmitted through the salt mass than through adjacent sedimentary formations. A. H. N.

271.* **Range and Application of Gas-Lift Methods. Part 4. Conclusion.** E. C. Babson. *Oil Wkly*, 25.12.39, 96 (3), 20.—The first cost of most intermitter installations is relatively small; emulsion trouble is usually not so severe as with straight gas lift, and the intermitter can be made to produce volumes of liquid as large as 2500 brl./day. Sub-surface mechanism of the intermitter, however, can seldom be repaired without pulling the tubing from the well.

If production from an intermitter is regarded as a series of slugs which flow into the filling-chamber, and are pushed one at a time up the tubing, the pressure held against the formation should equal the height of the slug multiplied by the density of the fluid. Available data indicate that the actual pressures are much higher than the results of such calculations. Methods of measuring these pressures are detailed. It is believed that intermitters are applicable primarily to the production of wells with medium static pressures and high productivity indices. Advantages of intermitters are described.

The gas-displacement pump is essentially an intermitter designed to function without a packer through the use of a full string of "macaroni" tubing hung inside con-

ventional tubing. The arrangement is considered as a separate method, and its characteristics and range of operation are studied in detail. It appears that the gas-displacement pump is directly competitive with sucker-rod and hydraulic pumps; but its application is limited to wells producing less than 200 brl./day.

A general discussion on all methods of gas-lift is given in this extensive paper. The various methods are compared against each other under similar conditions by graphical means. The relative economy of each method is evaluated graphically for certain conditions.

The paper ends with a discussion on the ranges of application of various gas-lift methods. It appears impossible to establish rigid ranges of application for the various methods, because the performance desired from a well is dependent on many variables, such as lease requirements, proration schedules, budget provisions, compressor facilities, demand for a particular grade of oil, and competitive conditions. However, in order to illustrate a point of view, some very generalized ranges of application—representing good practice on a large, relatively non-competitive lease—are shown in a diagrammatic form. The areas representing range of application are predicated upon a compromise between maximum production and minimum gas consumption, and not upon either factor alone.

The paper contains fifty-nine figures, many of which represent four charts each, each chart containing a number of interesting graphs. A. H. N.

272.* Predicting Wet Gas Recovery in Re-cycling Operations. J. S. Swearingen. *Oil Wkly*, 25.12.39, 96 (3), 30.—The discovery in recent years of a number of fields that yield a new type of petroleum production—the condensate or distillate type—has created new operating problems. Of particular importance at this time, due to the newness of such fields, is the estimation of ultimate yields, especially in view of the belief that it is advisable to instal re-cycling plants, which necessitates additional investment.

The prediction of ultimate recovery of condensate from a producing horizon where the residue gas is returned to the formation through another well is dependent on an estimate of the area displaced by the dry gas in this re-cycling operation, before the dry gas reaches the producing well. The problem is dealt with here as a study of two-dimensional displacement of the wet-gas layer by the injected dry gas. To show how rapidly the producing well may be expected to dry up after the dry gas reaches it, and what will be the shape and position of the dry-gas area, electrical models have been used, and the results are shown graphically in this paper.

A layer of electrolyte the ion content of which represents the gas content of the sand will respond to potential differences in exactly the same manner in which gas in the formation will respond to pressure differences. The electrical apparatus consisted essentially of half-cells representing wells and a layer of electrolyte solution on a glass plate, and made firm by a 2% agar content, the film representing the gas-saturated horizon. This agar solution was protected from drying by a covering sheet of thin mica. A small hole was drilled in the mica for each "well location." Numerous electrical models of fields of various shapes and well positions were made and tested, and successive patterns of the dry-gas area were made to scale. Details of the apparatus are given.

The results are given, and there appears at least one check on the method. It is understood that the Keeling well in the Long Lake field dried up approximately 9 months after a nearby re-cycling plant was put into operation. The application of the method checks the result.

It is contended that injection wells near the property lines are objectionable, as 30% or 40% of the dry gas may go under the adjoining lease. A. H. N.

273.* Petroleum-Reservoir Liquid Production Problems. K. Eilerts, R. V. Smith, and A. B. Cook. *Oil Wkly*, 1.1.40, 96 (4), 27.—This article is an abstract of the Bureau of Mines Report of Investigations, No. 3474. The summary of the full paper, of forty-seven pages and containing fifteen figures and fifteen tables, is as follows:—

Methods for determining properties of a reservoir fluid that are of particular significance to production practice have been developed. The equipment and procedure essential to the methods have been briefly described and the data obtained from a

typical reservoir fluid are presented. Practical applications of the data are illustrated, and suggestions are made regarding the additional information necessary for important extensions of the applications.

Apparatus developed include: a steel pycnometer for measuring the specific volume of fluids having high vapour pressures; a simplified variable-volume cell for determining the expansibility of fluids under conditions of phase equilibrium; and a fractional distillation assembly for analysing the gas and liquid hydrocarbon samples.

Analytical data are presented on a complete set of gas and liquid samples taken during a well flow-test. The composition and properties of a prepared reservoir fluid and three of its residua obtained by liberation of dissolved gas are given. Isotherms were determined for the four fluids in 30° F. intervals for the pressure range from 3000 to below 50 lb. per sq. in. abs.

The loss of energy from frictional effects was evaluated for two fluid production rates in each of three sizes of flow-string. Sub-surface pressures measured in a shut-in well off-setting a flowing well were applied to interpolation of the pressure gradients through the oil-producing structure at four rates of flow. The effect of changing fluid properties on the values of the gradients and the significance of the change in limiting withdrawal rates are discussed.

A development of particular interest to the petroleum technician is the analytical method described for comparing the compositions and other properties of hydrocarbon liquids obtained at different points in the flow-stream. The amount of gas liberated from liquid solution and the resultant volume change in the solvent can be determined without knowledge of the pressure and temperature gradients and the type of liberation in the flow-stream between the points at which the sample is taken. Production engineers are concerned with the decrease in energy that occurs as reservoir pressures decline, because the methods and rates of recovery depend on this factor. The energy available by expansion of reservoir fluids having compositions corresponding to three stages of depletion were estimated from experimental data obtained from residua of the original fluid.

A. H. N.

274.* Simple Dynamometer Card Analysis Method. S. B. Sargent. *Oil Wkly*, 1.1.40, 96 (4), 30-31.—The stresses and motions involved in sucker-rod pumping are treated mathematically as a problem in vibration. The method advocated in this short paper is simple, rapid, and appears to indicate a reasonable answer to the problem. Its value is, however, limited to wells where the tubing is anchored. In cases where the tubing is not anchored, a modification is suggested. This modification seems to be taking a dynamometer card on the tubing itself and adding vectorially the motion of the barrel to that of the sucker-rods or the plunger.

A. H. N.

275.* Limitations in Producing Wells with High Water-Oil Ratios. F. B. Taylor. *Oil Wkly*, 1.1.40, 96 (4), 43-44.—Wells in certain fields have to be abandoned despite the fact that they are potential producers of 50 or more brl./day, because to produce this quantity the operator has to lift from twenty to thirty times as much water. In cases like these abandonment is apparently the best method of storing the oil as a known major reserve to be drawn upon in future years, when reserves are less plentiful and production methods will be more efficient.

After reviewing cases actually occurring in such fields, it becomes evident that there are three major angles of approaching an economic solution:—

- (1) Possibility of reducing cost of operations.
- (2) Increasing fluid volumes.
- (3) Improving lifting means.

The first is limited, as neither labour, taxes, nor operating charges can be appreciably reduced. The second solution of increasing fluid volume provides a parallel decrease in high maintenance costs, and this trend is now under way. The third solution is exemplified by the use of submerged centrifugal pumps, hydraulic-actuated lifts, pneumatic lifting units, and rotary pumps which have given operators added leeway in many areas.

A. H. N.

276.* Heating and Dehydrating Natural Gas at the Well-Head. J. E. Moore. *Petrol. Engr.*, December 1939, 11 (3), 27.—Where well-pressures are high and temperatures low, the natural gas operator encounters problems that are intensified if severe climatic conditions characterize the area in which operations are conducted. Because whole communities are often entirely dependent on gas for heat, and because even brief interruptions of the gas supply would entail much suffering and inconvenience, the operator in such territories must know that the solutions found for these problems are correct in principle and are dependable in practice.

The author describes practices in two major gas-producing states of the North-west (U.S.A.), where gas is produced at elevations of 4000–8000 ft. above sea level, under pressures ranging upward to approximately 2500 lb./sq. in., and where winter temperatures sometimes fall to -50° F. These conditions cause peak-hour loads to run as high as twenty-five times the minimum summer loads.

To avoid the hazards of carrying extremely high pressures in the field gathering-lines, the gas pressure is reduced at the well-head. The resulting need for application of heat to counteract the refrigerating effects of expansion is satisfied by hot water or steam heaters, and not by direct heating, in order to ensure safety. Steam systems can meet variable demands more effectively. The peculiar advantages of each system are discussed.

The gas is cooled and the condensates are removed before delivery into the lead-lines. Particular attention is paid to removing all hydrates prior to the gas leaving the plant. Calcium chloride brine contactors are used.

A. H. N.

277.* Electric Submersible Well-Pump Performance. W. H. Stuove. *Petrol. Engr.*, December 1939, 11 (3), 38.—Although this paper deals with energy consumption and lifting costs specifically for the St. Louis Pool, Oklahoma, it is believed that the results will have a more general application to other pools. The production is from an average depth of 4100 ft. by about 800 wells, sixty-one of which are equipped with electric submersible well-pumps. The first of these pumps, of 97½-h.p. capacity, were tried out in this field in 1934. Fluid was lifted at the average rate of 1700 brl./day, with an average energy consumption of 1.35 kw.hr./brl., and an overall efficiency from wire to fluid in the surface tanks of about 45%. The average oil recovered was approximately 9% of the total fluid pumped when considered over a period of years for all pumps of this type in use.

Tables and graphs are supplemented by analysis of the problems. It is found that the conditions in many wells necessitate pumping at as high a rate as possible in order to obtain profitable operations. Many large-capacity wells, when producing at the rate of about 400 brl./day, yield only water and a negligible amount of oil, yet, were the same wells pumped at a rate of 1400–1700 brl./day or greater, the percentage yield of oil to water would increase by as much as 10%.

A. H. N.

278.* Scrap Metal Abandoned by the Oil Companies. A. P. Burguillos. *Rev. del Min. Fomento (Venezuela)*, July 1939, II (14), 83–105.—The author discusses the feasibility of utilizing the large quantities of scrap metal abandoned by the oil companies at different places where they have been operating. He suggests that it would be worth while to start works equipped for the manufacture of small implements and tools, such as picks, shovels, hoes, and the like. In order to carry out such a scheme it would be necessary for the State to restrict the re-export of scrap materials, and especially those which were originally imported free of duty.

The general legal aspect is examined and the opinion expressed that any person prepared to pay the cost of transport would have the right to remove and utilize the materials scrapped. It is suggested that the proposal is worth consideration in view of the marked rise in the cost of materials, especially metals, due to the immense amount of rearmament taking place at the present time.

H. I. L.

279.* Phase Diagram of a Mixture of Natural Gas and Natural Gasoline Near the Critical Conditions. D. L. Katz, D. J. Vink, and R. A. David. *Petrol. Tech.*, November 1939, A.I.M.M.E. Tech. Pub., No. 1114.—A phase diagram showing boundary curve and quantity of liquid in the two-phase region was determined for a mixture of natural gas and natural gasoline in the regions of its critical conditions. The temperature and

pressure of the phase measurements were in the range of 85–212° F. and 1300–2600 lb./in.², respectively, with the critical condition at 169.5° F. and 2615 lb./in.² absolute.

The pressure drop required to change the system from a uniform phase to a definite percentage of liquid became smaller as the temperature was raised above 85° F. Striking colour phenomena accompanied the measurements near the critical temperature. At pressures considerably above the two-phase region the system was colourless. As the pressure was lowered towards the bubble or dew point the single phase took on a reddish colour. At temperatures near the critical the colour was a bright mahogany red, but a faint colour was perceived over the entire range 102–192° F.

No unusual change was observed in the shape of the pressure-volume curves for the two-phase system in passing from temperatures below the critical temperature to the region of retrograde condensation. The plot of saturated density appears to be a smooth curve for the bubble-point liquid below the critical temperature and for the dew-point vapour above the critical temperature.

G. D. H.

280. Production Patents. L. F. Rollins. U.S.P. 2,185,522, 2.1.40. Appl. 28.10.37. Well-flow controlling device consisting of perforated section in a casing positioned at flow strata and plugs for filling the perforation to shut off the flow of fluids through these perforations and movable means for holding the plugs in place.

E. W. Patterson. U.S.P. 2,185,680, 2.1.40. Appl. 17.6.36. Well-pumping mechanism using a sampson post, walking beam and pneumatic balancing apparatus creating and maintaining a constant air pressure.

M. A. Elliott. U.S.P.P. 2,185,725 and 2,185,726, 2.1.40. Appl. 12.5.38 and 21.12.38. Adapter coupling for joining rigid and flexible pipes, the latter for pipes of different diameters.

E. S. Semmons. U.S.P. 2,185,727, 2.1.40. Appl. 3.8.36. Hydraulic pump.

J. Preston. U.S.P. 2,185,925, 2.1.40. Appl. 29.9.38. Oil-well oil saver.

A. H. N.

Transport and Storage.

281.* Review of the U.S. Bureau of Standards Work during 1939 on Underground Corrosion. K. H. Logan. *Oil Gas J.*, 21.9.39, 38 (19), 112.—Recent investigation by the National Bureau of Standards on soil corrosion has been along the three following lines: (1) analyses of soil corrosion data provided by pipe-line operators; (2) development of tests and methods of measurement which may be of immediate practical value to corrosion engineers, and (3) studies of corrosion processes.

1. Statistical surveys of the average physical condition of pipe-lines are made, based on the average depth of the deepest corrosion pits. These are correlated with tests on the corrosivity of samples of soil adjacent to the pipe, but present indications are that the correlation is not close. If a considerable number of samples are taken, however, a rough indication of probable corrosion can be obtained.

2. The determination of the potential required for adequate cathodic protection of lines is frequently based on the copper sulphate electrode or half-cell, but this may give misleading results, owing to variations in the purity and hardness of the copper electrodes, aeration, light, and temperature. These factors have now been studied, and suitable corrections are available. It is also recommended that they should only be used with indicating instruments for which they were designed or with potentiometers, since with indicators of low resistance, polarization and other causes give erroneous results. An improved type of copper cell has been devised, and it is felt that the voltage and current density required for cathodic protection can now be determined. Experimental work on operating lines will be put in hand when the laboratory work is completed.

3. The most promising test of soil corrosivity is considered to be the use of an electrolytic cell in which the current flows from the anode through the soil to a cathode of the same material as the anode, but kept at a different potential by differential aeration. The loss of weight in the anode indicates the corrosivity of the soil. Indications of this are also given by the change in potential of the electrodes as the current is

increased by means of superimposed voltages, the measurements being made while the current is interrupted, for which purpose mechanical or electronic methods have been devised. Loss in weight of anode is also in line with the order of current density at the corrosion potential (potential at which current density-potential curves intersect), and it may be possible to correlate soil corrosivity with the maximum short-circuit current in the corrosion cell. For many soils the rate of corrosion is determined primarily by the rate of the cathode reaction, but in some cases marked polarization of the anode takes place, indicating the formation of protective films. The loss of protective coatings of corrosion products by e.g. diffusion in marshy soils is studied in glass cells in which the movement of membranes, formed by reaction between ferrous ions diffusing towards the cathode and hydroxyl ions and oxygen moving towards the anode, could be followed. Membrane development at appreciable distances from the anode is associated with a high rate of corrosion, whereas in slightly acid or alkaline soils it is found in contact with the anode, corrosion being thereby partly inhibited.

C. L. G.

282.* Determining Minimum Current Required to Provide Adequate Cathodic Protection. Part I. D. Harrell and M. Clerk. *Petrol. Engr.*, October 1939, **11** (1), 38-44.—In applying cathodic protection the negative potential at which corrosion ceases is of a definite magnitude, so that any excess is a waste of power. The loss-of-weight method hitherto applied has the disadvantage of measuring the maximum safe limit and not the more economic safe limit of the potential. The method evolved for determining the minimum potential has been arrived at from a consideration of the phenomena observed in the application of cathodic protection—viz., deposition of metallic iron, increase of soil alkalinity and evolution of hydrogen at the cathode (pipe) and a corresponding increase of corrosion, evolution of acid-radicle gases, and increase of acidity at the anode. From a consideration of these facts an evaluation of the minimum protective potential has been deduced. It is shown that this potential is characterized by (1) a decrease of the ferrous iron concentration at the cathode to virtually zero, (2) a sharp increase of hydroxyl concentration at the cathode, (3) total ionic concentration reaches a minimum at which point hydroxyl-ion concentration equals the ferrous-ion concentration, (4) decomposition of the water and evolution of hydrogen begins, (5) a further increase of potential effects little reduction of ferrous ion, the energy inducing the evolution of hydrogen.

R. J. E.

283.* Distributed Anode Method Cuts Cost of Cathodic Protection. O. C. Roddey and L. R. Sheppard. *Oil Gas J.*, 21.9.39, **38** (19), 84.—Investigations on the prevention of electrolytic corrosion have been carried out by the Interstate Natural Gas Co. using as laboratory a portion of line in Tensas Swamp La. selected in August 1932. It has been shown that the installation of ground beds at 50 and 100 ft. and to a smaller extent at 700 ft., using 200 amps. at 47 volts, gave excessive potentials directly opposite the units, whereas a few hundred feet down the line there was barely enough to prevent corrosion. A system of distributed anodes was devised using anodes carrying much less current (50 amps. at 3.3 volts) at 37-50 ft. intervals located 25 ft. from the line. Power requirements for successful protection of pipe, using copper oxide rectifiers and distributed anodes, are given as: 13.1 amp. per mile for 22-in. pipe hot enamel-coated in 1926; 8.3 amp. per mile for 14-in. pipe asphalt coated and wrapped with roofing felt in 1925, and 8.0 amp. per mile for 13-in. pipe hot enamel-coated in 1926. The anodes, consisting of 2 × 80 in. plain electrode furnace carbon impregnated with hot paraffin, have shown very little increase in resistance over 5 years service, and should theoretically last 50 years.

C. L. G.

284.* Microbiological Anaerobic Corrosion of Steel Pipe-lines. R. F. Hadley. *Oil Gas J.*, 21.9.39, **38** (19), 93.—A review is given of research on the corrosion of steel pipe-lines by anaerobic sulphur-reducing bacteria, and of the examination of corroded pipe-lines in swampy areas with reasonably high soil resistivity. The presence of ferrous corrosion products on pipe in water containing sulphate indicates bacterial reduction. The conditions required for bacterial corrosion activity were found to be: (1) the presence of air-tight or water-saturated ground with little motion of the water, (2) a pH value of 6.2-7.8, and (3) the availability of organic food. The primary strain of sul-

phur-reducing bacteria (*Vibrio desulphuricans*) has been shown to be relatively insensible to wide temperature ranges (0–80° C.), and, being in size 2μ to 5μ long by 0.5μ wide, are readily visible under a microscope. The process of attack postulates the utilization by the bacteria of the hydrogen of polarization from cathodic areas in the reduction of the combined oxygen of the sulphate ion forming H_2S and water. The ferrous ions are precipitated by the hydrogen sulphide as ferrous sulphide and by the hydroxyl ions as ferrous hydroxide, thus lowering the solution pressure of the ferrous ions and accelerating the corrosion process owing to the removal of the cathodic hydrogen polarization. The presence of ferrous sulphide on a corroded pipe is a reliable indicator of the presence of sulphur-reducing bacteria. Corrosion by this means is of abnormal intensity, and cathodic protection against it is of doubtful value. This problem is, however, being studied.

C. L. G.

285.* Cathodic Protection and Polarization of Underground Pipe-Lines. R. J. Kuhn. *Oil Gas J.*, 21.9.39, 38 (19), 98.—The importance of the protection against further corrosion afforded by the hydrogen formed by polarization of galvanic couples in pipe-lines is discussed and the factors affecting polarization are outlined. The current discharged from the pipe at anodic spots of corrosion collects on cathodic areas of the pipe accompanied by a deposit of hydrogen on the surface of the pipe and by the formation of a layer of alkaline earth, due to the removal of the acid hydrogen ion from the adjacent earth. The rate of corrosion is checked by the resistance effect of the hydrogen and by the change in potential owing to the hydrogen coating. In dry areas the hydrogen may be quickly removed by atmospheric or dissolved oxygen, whereas in wet areas it may be retained, so that the state of the soil has a great bearing on the rate of corrosion. Reconditioning of an old line or installation of a new line will frequently cause very rapid corrosion to take place on the new section, owing to the removal of protective films and to potential differences between the old and new lines. Similarly corrosion test-holes are liable to undergo rapid subsequent corrosion. Depolarization may also be caused by oxygen-producing bacteria. The potential applied to give cathodic protection may also be reduced by the formation of a film of hydrogen on the areas of the pipe on which cathodic currents are collecting.

C. L. G.

286.* Method for Applying Thick Asphalt Mastic Coating. N. W. Colley. *Oil Gas J.*, 21.9.39, 38 (19), 150.—A description is given of a rapid method developed in California for the application *in situ* of a dense asphalt mastic coating to pipe-lines. After ditching and raising the pipe, it is cleaned by a travelling cleaning machine equipped with scrapers, knives, and cutters, and sand-blasted in two stages, pits being subsequently spot welded and the pipe patched where necessary. A primer is then applied by two pressure spray-guns, and covered under pressure with a seamless uniform coating of asphalt mastic, consisting of a dense, carefully graded mixture of sand, limestone dust, asphalt, and asbestos. The latter is manufactured in a mobile hot plant and applied by a special travelling expanding nozzle to a thickness of $\frac{1}{2}$ in. at speeds varying from 8 to 14 ft. per minute. The nozzle is equipped with expanding quadrants, which allow for coating over patch plates, high welds, etc.

A similar process is in use at railhead and central plant operation for the coating of new or reconditioned pipe. Reconditioning is effected by first cutting out welds and collars, straightening and then heating in a furnace, where internal residues and external paint, etc., are removed. It is then heated to an annealing temperature to remove internal stresses, gradually cooled, and the pipe-ends are bevelled for subsequent welding. Finally, it is cleaned with steel grit in a roto shot-blast machine, pits and thin spots are built by electric welding and any roll welds made, and, after testing, coated with primer, asphalt mastic, and whitewash.

C. L. G.

287.* Location and Installation of Ground Beds. D. F. Ball. *Petrol. Engr.*, October 1939, 11 (1), 91–94.—The selection of test instruments for the measurement of soil resistance is discussed and the A-C fall-of-potential method recommended. The use of a modified version of this method for surveying an area to select the site of the ground-bed is described. The relative merits of vertical and horizontal ground-beds are dependent on conditions. The former has the disadvantage of passing through a number of soil strata which may vary in resistance, whilst the resistance of the latter is

subject to seasonal variations. To obtain good pipe-to-soil contact the use of sodium or calcium chloride is recommended, and the method of applying this treatment, effectively, is described.
R. J. E.

288.* Ten-Year Trial Shows Success of Inert Gas System. M. G. Van Voorhis. *Nat. Petrol. News*, 18.10.39, 31 (42), R. 441.—The displacement of combustible gases in storage tanks, refinery vessels, and in tankers by inert flue gases as a fire preventative and for the production of safe working conditions is a system which has proved to be notably effective. A number of small generating plants are distributed at strategic points about the refinery, adequate safety measures, especially where the units are on board tankers, being taken. The purging process is normally controlled and its progress followed by means of gas analysis by the Orsat method.
H. G.

Gas.

289.* Cost of H₂S Removal from Refinery and Sour Natural Gas. B. Storrs. *Oil Gas J.*, 19.10.39, 38 (23), 69.—Treating data are presented on three Girbotol plants for the removal of H₂S from (a) 5000 cu. ft. per day of stabilizer gas containing 5% of H₂S, reduced to max. 30 grains per 100 cu. ft.; (b) 6700 cu. ft. per day of stabilizer gas containing 300 grains H₂S per 100 cu. ft. reduced to max. 10 grains per 100 cu. ft., and (c) 8000 cu. ft. per day of natural gas containing 163 grains per 100 cu. ft., the plant being subsequently modified to effect simultaneous dehydration and purification. For this purpose a solution containing 11% monoethanolamine, 72% diethylene glycol, and 17% water was used. The total make-up amounted to 0.13 lb. monoethanolamine and 0.30 lb. diethylene glycol per million cu. ft. of gas, costing 7 cents per million cu. ft. The total treating cost varies from less than 0.5 c. per 1000 cu. ft. for natural gas with a low H₂S content to 2 c. per 1000 cu. ft. for refinery gas of unusually high H₂S content. The presence of CO₂ in the gas also increases the overall cost, owing to its absorption by the ethanolamines.
C. L. G.

290.* Apparatus for Study of Pressure-Volume-Temperature Relations of Liquids and Gases. B. H. Sage and W. N. Lacey. *Petrol. Tech.*, November 1939, A.I.M.M.E. Tech. Pub. No. 1127.—An apparatus is described for the measurement of the pressure-volume-temperature relations of pure substances, simple mixtures, and complex mixtures with an over-all absolute uncertainty, which is usually not more than 0.2%. The equipment is suitable for studies in the gaseous, two-phase, and liquid regions. The behaviour of such systems may be investigated at pressures as high as 10,000 lb./in.² for temperatures between 70° and 600° F. The methods of measurement and the construction of the apparatus are described in some detail.
G. D. H.

291.* Volumetric Behaviour of *iso*Butane. W. M. Morris, B. H. Sage, and W. N. Lacey. *Petrol. Tech.*, November 1939, A.I.M.M.E. Tech. Pub. No. 1128.—The volumetric behaviour of carefully prepared *isobutane* was investigated over a temperature range of 100–460° F. and at pressures up to 5000 lb./in.² At pressures in the neighbourhood of 700 lb./in.² at 280° F. the actual volume of *isobutane* was only about 20% of that predicted from the perfect gas law, but at pressures over 4500 lb./in.² the specific volume was greater than that predicted from the perfect gas relationship.

The data do not overlap other published information in the gaseous region.

G. D. H.

Cracking.

292.* Reaction Rates in Cracking. W. L. Nelson. *Oil Gas J.*, 26.10.39, 38 (24), 132.—Data on the cracking reaction velocity constants of a series of hydrocarbons and of various petroleum fractions have been correlated to provide a chart showing the variation of this constant with temperature. Most of the data were taken from tests conducted mainly to determine the type of reaction rather than the rate, and it has been necessary to disregard a number of factors for the sake of a generally available correlation. The chart does not indicate the exact products of decomposition, and

should not be used for cracking yields larger than 25%. The effect of pressure is disregarded as being of little significance except in extreme cases. Difficulty is also caused by the fact that decomposition is not the only reaction taking place during cracking, so that material heavier than the charge stock, as well as pure decomposition products, are obtained. The decomposition reaction is defined by the following

formula: $-K_1 = \frac{1}{t} \log_e \left(\frac{a}{a-x} \right)$, where K_1 is the reaction velocity constant (yield per second); t is the time in seconds; a is the percentage of material being decomposed that is present in a feed-stock (thus $a = 100$ for a pure feed-stock), and x is the percentage of feed material disappearing in the reaction during time t .

Examples of the use of this chart are provided.

C. L. G.

Polymerization.

293.* **Polymerization of Hydrocarbons Present in Natural Gas.** R. Fussteig. *Petrol. Engr.*, October 1939, 11 (1), 219-223.—The various methods available for the polymerization of the lower paraffin hydrocarbons present in natural gas are described. The first stage in the polymerization of these hydrocarbons is dehydrogenation or demethanization to gaseous olefines. The results of the thermal decomposition of individual hydrocarbons are described. Two methods are applied to convert the paraffins into olefines: thermal and catalytic. Catalysts are of two types, one of which effects dehydrogenation and the other demethanization. Detailed processes commercially applied to polymerize hydrocarbons are given, together with flow-sheets. Some of these processes involve polymerization of olefines only, whilst in others decomposition of paraffins to olefines and polymerization of the latter are combined. Catalytic polymerization may be selectively applied—e.g., the butane-butylene fraction of cracked gasoline may be removed by deep stabilization and subjected to polymerization to give diisobutylene, which is subsequently hydrogenated to iso-octane.

R. J. E.

Synthetic Products.

294.* **Petrol Synthesis. Temperature Control in the Direct Process.** Anon. *Chem. Tr. J.*, 3.11.39, 105, 347.—Optimum conditions of temperature, rate of throughput, contact time, and pressure for the production of good yields from the synthesis of carbon monoxide and hydrogen are described by the Metalgesellschaft of Frankfurt in E.P. 507,366 of 1938. In an example given, gases containing 2 vols. of H_2 to 1 vol. of CO are reacted at 180° C. and normal pressure, the throughput being 1 litre per hr. per gm. of cobalt in the contact mass, the contact time 36 sec., and the heat-load per sq. in. 240 kg.-cals. per hr. A yield of 100 gm. per c.metre of gas was obtained, a similar yield being given on using a chamber twice the capacity and the same amount of catalyst diluted to twice the volume, increasing the throughput to 1.4 litres and the contact time to 51.5 sec., the temperature to 185° C., and reducing the heat load to 168 kg.-cals. A catalyst containing 33% cobalt, 6% thoria, and 61% kieselguhr is used, it being stated that dilution of the catalyst with kieselguhr to a content of 2% cobalt gives a material particularly suitable for the synthesis of lower hydrocarbons from benzene to gasoil.

C. L. G.

295.* **Hydrocarbon Synthesis from Carbon Monoxide and Hydrogen over Alloyed Catalysts.** I. Rapoport and E. Polozhintseva. *Oil Gas J.*, 28.9.39, 38 (20), 52.—The results of investigations of the use of alloyed Ni-Al, Co-Al, and Co-Si catalysts in the synthesis of hydrocarbons from CO and H_2 are presented. The Ni-Al catalysts were prepared in the form of (a) lentils and (b) powder, a yield of 140 c.c. of hydrocarbons per c.metre being the highest figure obtained when using a lentil Ni-Al catalyst at 170° C. Lower yields were given by the powdered catalyst, by the introduction of NH_3 , and by increasing the temperature or the volume velocity. At the same volume velocity it is necessary to use Co-Al (powder) catalyst at a higher temperature (200° C.) in order to attain similar yields, but at half the volume velocity and at 200° C. a yield of 175 c.c. of liquid hydrocarbons per c.metre is obtained. With the Co-Si

catalyst, lower yields (max. 109.6 c.c.) were given oven at 220° C., and with much lower volume velocities. This catalyst also appears to give a greater yield of solid paraffins.
C. L. G.

296.* **Photochemical Reactions under High Pressure.** K. Peters. *BrennstChemie*, 1939, 20, 356-358.—A quartz-mercury lamp was placed in a cylindrical pressure vessel of 50 mm. inside diameter which could be tilted to permit ignition. Lamps were resistant to pressures up to 80 atm., but making lamps for use under several hundred atmospheres would present no difficulties. Preliminary tests were carried out under pressures up to 50 atm. with CO-H, CO-H-NH₃, CO₂-H, CH₄-CO₂, and CH₄-O₂. In all tests where water was either present or formed during the reaction, excessive rust formation of all iron components within the pressure vessel, especially of the finely polished wall, was observed. As aluminium was also subject to corrosion, a silver coat is recommended.

In all tests chemical reactions were observed, although mostly small in extent. With CH₄-O₂ mixtures containing 2-3% O₂, the oxygen was completely consumed after a few minutes' illumination. However, in the reaction gas neither CO nor CO₂ could be found in quantities corresponding to the oxygen volumes consumed. L. R.

297.* **Effect of CO-H on Cresol under Pressure.** W. Krönig. *BrennstChemie*, 1939, 20, 355-356.—When *m*-cresol together with CO-H are passed, under 200 atm. pressure at 500° C., over methanol-forming catalysts, the cresol, besides being reduced to hydrocarbons, is also methylated, most probably to trimethylphenol. L. R.

Refining and Refinery Plant.

298.* **Use of Gum Inhibitors in Refining of Motor Fuels.** W. W. Scheumann. *Oil Gas J.*, 2.11.39, 38 (25), 41.—Modern gum inhibitors have been selected from compounds containing phenolic or primary or secondary aromatic amine groupings, which, in addition to preventing deterioration of the gasoline, have the following characteristics: it should be soluble in gasoline, insoluble or very sparingly soluble in water or NaOH solutions, it should not impart colour to the gasoline, and should be non-reactive with tetraethyl lead or oil-soluble dyes. Those in general use to-day include such materials as the monobenzyl, dibenzyl, *isopropylmonomethyl*, monobutyl and *monoisobutyl* derivatives of *para*-aminophenol, as well as hardwood tar acids.

Gum inhibiting may be coupled with the following treating methods:—

1. Production of end-point and vapour-pressure material directly from the unit and sweetening.
2. Clay-treating end-point material to polymerize only the most unstable constituents and sweetening.
3. Using a light acid treatment on a benzine fraction followed by re-distilling and sweetening.
4. Splitting a benzine fraction into a light and heavy portion, acid-treating and re-distilling the heavy fraction, and sweetening the combined re-distilled heavy gasoline and the light gasoline.

Method 1 is valuable where the equipment is available and where a water-white final colour is not required, as when dyes are used. It is estimated that a possible saving of 3 to 4 cents per barrel of gasoline of a cracked Mid-Continent type can be effected owing to the elimination of the cost of refining, and of the volumetric and octane number loss on treating.

Method No. 2 cheapens the clay treatment by enabling a product of poorer initial colour and induction period to be produced, these characteristics being improved by subsequent inhibiting. Method No. 3 similarly reduces acid costs and volumetric and octane number losses.

Method No. 4 (which is described in detail) represents a saving of 2 cents per brl. of gasoline over the conventional method of acid-treating and re-distillation.

Careful control over sweetening before inhibiting is necessary to prevent any free sulphur lowering the inhibitor susceptibility, and to remove any traces of alkali which

may react with the inhibitor. In these directions processes such as copper-sweetening offer obvious advantages. The relation between improvement in induction period and inhibitor concentration should be studied when deciding on the most effective inhibitor of those available for the particular gasoline to be treated. Figures are given showing the relative costs of inhibiting two gasolines with four different inhibitors, indicating the necessity for careful selection of the inhibitor. C. L. G.

299.* Motor Fuels Refinery is Designed for West Texas Crude. D. H. Stormont. *Oil Gas J.*, 28.9.39, 38 (20), 46.—A description is given of a refinery near Levelland, West Texas, designed to handle West Texas sulphur crude, and consisting essentially of a Dubbs combination topping and cracking unit with a straight-run capacity of 4500 brl. daily and a cracking capacity of 3000 brl. daily. The plant is very flexible and has a high degree of instrumental control.

The crude is settled at 225–250° F. to remove salt, etc., before processing. The cracked gasoline is stabilized and treated with caustic to remove mercaptans, dilute acid to remove nitrogen compounds, dried with activated alumina, and sweetened with copper sulphate and ammonium chloride. A further treatment with zinc sulphide is given to remove any traces of copper, and it is then inhibited with U.O.P. No. 4 inhibitor.

The Slaughter field crude used yields 34.6% gasoline and 9.3% kerosine. The gasoline has an end point of 400° F. distilling 27% to 212° F. and an O.N. of 56.5 and sulphur content of 0.28%; cracking of the reduced crude yields 42% of cracked gasoline. C. L. G.

300.* Distillation Process of Natural Gasoline Extraction. R. W. Machen. *Petrol. Engr.*, October 1939, 11 (1), 96–97.—The distillation of natural gasoline has the object of fractionating the rich oil to give the desired gasoline fraction and reducing the oil to as near as possible its original composition for recycling through the absorber. The rich oil passes through heat exchangers into the evaporator, where the bulk of the gasoline plus a small part of the light fractions of the oil are immediately flashed and passed to the bottom of the rectifying column. In the rectifying column the vapours rise from tray to tray in such a manner that intimate contact is obtained at each tray between the rising vapours and descending stream of liquid. The liquid at successive trays becomes richer in lighter components and poorer in heavy constituents, whilst the descending oil becomes heavier and poorer in volatile fractions as it falls to the base of the evaporator, whence it is ultimately returned to the absorbers for recycling. Normal condensation is insufficient to maintain the continuous downward liquid flow, and condensate is introduced at the top of the column as reflux. The quantity of reflux depends on the separation desired and the end-point required for the gasoline. Steam is introduced into the still, the amount depending on the stripping desired. By lowering the partial pressure of the hydrocarbon gases, steam permits the stripping of the heavier gasoline fractions from the oil and increases efficiency by increasing the evaporating surface in the agitation of the oil. R. J. E.

301.* Review of Roumanian Petroleum Refineries. V. Cerchez. *Monit. Petr. roum.*, 1939, 40, 1061–1065.—This article consists of a very complete review of Roumanian refineries tabulated according to the enterprises operating in the country. Very full details are given of the operation of the major companies during 1938. The refineries are divided into two groups: those with and those without cracking facilities. The former group accounts for about 80% of the industry.

In the following details, all figures relate to annual capacity in tonnes or to throughput or treatment during 1938.

Astra Romana, capacity 2,000,000, treated 1,492,504. These refiners, the biggest in Roumania, possess a battery of twelve crude stills capable of 1,000,000 throughput which are operated to 50% capacity. The tube-stills (McKee system) handled 968,040, or about 97% capacity. The lubricating-oil stills dealt with 75,778 of an 83,000 capacity.

The cracking installation consists of four Dubbs units which operated to about 80% capacity in treating 516,816. The pressure distillate is processed in a Trumble plant.

White spirit, kerosine, etc., are handled by ten agitators, which were kept practically at 100% capacity, but the lubricating-oil refinery plant, which possesses three Laval centrifuges, handled only 35% of its maximum.

The cracked-spirit stabilization plant treated 174,757 and the tetraethyl lead plant 2587. Production of liquefied gas was 2191, of naphthenic acids 504, of bitumen 31,010, and of light petroleum solvents 1005.

A reforming plant is under construction. Storage consists of 334 tanks with a capacity of 424,501 tonnes. There are 1781 employees.

The Romano Americana refinery has a capacity of 1,400,000 and treated 898,054. The crude distillation is dealt with by a Standard-Oil tube system (operating to 80% capacity) and by a battery of eight stills operating to about 50% capacity. The lubricating-oil distillation plant throughput was 23,533, against a maximum of 70,000. The refinery also possesses a Foster Wheeler tube system, which handled 61,000, or a little over 30% of its capacity, and three stills capable of continuous or batch operation which did not operate in 1938. The company also operate a Tube and Tank cracking system which handled 363,952, or 91% of capacity. Kerosine refining is handled by two agitators of 240,000 capacity, which have been used to a little over 50% capacity.

Gasoline is treated in a continuous system, which ran to about 50% of its 200,000 maximum.

Although the distillation plant produced 23,000 of lubricating oil, only 4979 were refined. The lubricating-oil refining plant, which operated to about 11% capacity, consists of two acid and three soda agitators.

Among the miscellaneous equipment of the refinery is an ethyl blending plant and an air-blowing plant for bitumen. 9510 tonnes of bitumen were produced in the year under review.

Storage consists of 149 tanks with a total capacity of 163,957 tonnes. There are 1396 employees.

T. C. G. T.

302.* General Review of the Roumanian Petroleum Refineries. V. Cerchez. *Monit. Petr. roum.*, 1939, 40, 1189-1193.—A further number of Roumanian refineries are reviewed.

The Aquila refinery of the Colombia company has an annual capacity of 540,000 tonnes of crude, which in 1938 treated 285,614 tonnes. The plant consists mainly of a Winkler-Koch tube system coupled with a Winkler-Koch cracking installation. Storage consists of seventy-one tanks with a total capacity of 64,750 tonnes, and the personnel numbers 350.

Creditul Minier de Brazi is the newest refinery in the country, having come into operation in 1935. Its annual capacity is 540,000 tonnes, and in 1938 it handled 389,410. This refinery has a Brunn Konigsfeld tube system and a Dubbs cracking plant. Their storage consists of sixty-one tanks, holding 72,350 tonnes. Personnel amounts to 430.

The Petrol Block refinery Standard do Ploesti has a capacity of 504,000 tonnes. It has a battery of nine continuous stills, which in 1938 treated 126,864 tonnes of their 360,000 capacity. A second battery of seven stills has been idle. The refinery has also a Dubbs cracking plant of 144,000 tonnes capacity, which handled about 53,000 in the year under review. Storage capacity is 82,058 tonnes and personnel 330.

T. C. G. T.

303.* General Review of the Roumanian Petroleum Refineries. V. Cerchez. *Monit. Petr. roum.*, 1939, 40, 1305-1309.—The extensive review of the Roumanian petroleum refineries in 1938 is continued. In this section the author deals with a group of relatively small refineries which do not possess cracking equipment.

The refineries and their annual capacities and throughputs in 1938 are: Xenia (Ploesti)—capacity 292,000 tonnes, throughput 137,342 tonnes; Dacia Romano, 240,000 and 119,695; Petrolul Bucuresti (Prahova), 210,000 and 187,170; Lumian (Petrolmina) Ploesti, 150,000 and 41,254.

The average personnel in these installations is about 125 and the storage capacity 30,000 to 50,000 tonnes.

T. C. G. T.

304.* Waste Problems in the Petroleum Industry. J. B. Hill. *Industr. Engng Chem.*, 1939, **31** (11), 1361.—This paper describes modern methods for the disposal of waste products in the petroleum industry.
H. E. T.

Chemistry and Physics of Petroleum.

305. Rearrangement of Certain Derivatives of Toluene by the Action of Aluminium Chloride. J. F. Norris and H. S. Turner. *J. Amer. chem. Soc.*, 1939, **61**, 2128–2130.—The authors have studied the effect of heating with aluminium chloride in the presence of hydrogen chloride *o*-, *m*-, and *p*-chlorotoluene, *o*-, *m*-, and *p*-nitrotoluene, *p*-cresol, and *p*-dimethylaminotoluene. In some cases the effects of change in time, temperature, and molecular ratio of the aluminium chloride to the organic reactant were studied. They found appreciable rearrangement and disproportionation with the chlorotoluenes, the extent being determined by the conditions under which the reaction proceeded, as mentioned above. Under certain fixed conditions they found that the order of the stabilities of the isomers were *ortho*, *meta*, *para*; and also that the mixture of the isomers obtained from *p*-chlorotoluene consisted of *ortho*, *meta*, and *para* in the ratio 1 : 2 : 1. In the same conditions the isomeric nitrotoluenes and *p*-dimethylaminotoluene were unaffected. It appeared that the *ortho* and *para* directing groups, methyl, hydroxyl, and chlorine, produced in a methyl radical sufficient reactivity to permit a change of position of the latter in the molecule, whereas the *meta* directing nitro-group stabilized the methyl radical so that it did not migrate under the conditions that affect the other class of compounds.
R. D. S.

306. Rearrangement of the Xylenes by Aluminium Chloride. J. F. Norris and G. T. Vaala. *J. Amer. chem. Soc.*, 1939, **61**, 2131–2134.—A detailed study of the rearrangement of the xylenes is reported, including investigations into the effects of time, temperature, molecular ratio of hydrocarbon to aluminium chloride, presence of ferric chloride in varying amounts, and presence or absence of hydrogen chloride. Change in temperature affected the ratios in which the isomers were formed, and decrease in the molecular ratio of aluminium chloride decreased the rate of rearrangement and percentage decomposed. The presence of ferric chloride had a negligible effect, and the presence of hydrogen chloride apparently increased decomposition, but did not affect rearrangement. Rearrangement was more rapid than the decomposition of the hydrocarbons and yielded a mixture approaching the composition of commercial xylene. The authors suggest that the results quoted may be employed in the preparation and the elucidation of the structures of compounds obtained by the Friedel-Craft reaction.
R. D. S.

307. Oxygen Effect in the Reaction of cycloPropane with Bromine and with Hydrogen Bromide. M. S. Kharasch, M. Z. Fineman, and F. R. Mayo. *J. Amer. chem. Soc.*, 1939, **61**, 2139–2142.—*cyclo*Propane and bromine in the ratio 10 : 1 reacted very slowly even in the light in the absence of oxygen, but the presence of oxygen greatly accelerated the reaction, and organic peroxides exerted a similar catalysis. Ethyl alcohol and *iso*amyl nitrite inhibited the photochemical oxygen-catalysed reaction. Only 1 : 3-dibromopropane was isolated from the reaction as small quantities of reactants were used, but there was evidence that other products were formed. With hydrogen bromide *cyclo*propane was used in tenfold excess, and also in equimolecular proportions. With excess of the hydrocarbon, oxygen tremendously increased the reaction rate, which was otherwise very slow, even in the light. Normal propyl bromide was the product. Benzoyl peroxide again had a similar effect to oxygen, and antioxidants again inhibited the reaction. Oxygen and light, however, had little effect on the reaction between equimolecular proportions of *cyclo*propane and hydrogen bromide, although compounds containing the hydroxyl group accelerated it. For the reaction between *cyclo*propane and bromine, and between excess *cyclo*propane and hydrogen bromide, the authors put forward a chain mechanism in explanation, but consider that the mechanism of the reaction of *cyclo*propane with hydrogen bromide in equimolecular proportions is ionic in character.
R. D. S.

308. Chlorinations with Sulphuryl Chloride. I. The Peroxide-Catalysed Chlorination of Hydrocarbons. M. S. Kharasch and H. C. Brown. *J. Amer. chem. Soc.*, 1939, **61**, 2142-2150.—The use of sulphuryl chloride as a chlorinating agent has hitherto depended on the production of molecular chlorine during the reaction, so that there has been little advantage over gaseous chlorine in its employment. The authors have discovered that in the presence of certain catalysts (such as organic peroxides) sulphuryl chloride can serve as a source of atomic chlorine, with the result that they have been enabled to chlorinate in the dark paraffin hydrocarbons, side-chains of aromatic compounds, aliphatic acids, aldehydes, and ketones, etc., far more rapidly and conveniently than is possible by the use of elementary chlorine in the presence of light. The best catalyst was found to be benzoyl peroxide; the less stable lauroyl peroxide was also found to be useful in some cases. The present communication is concerned with the chlorination of hydrocarbons, the results obtained being generally similar to the photochemical chlorination of these compounds by chlorine gas, as the authors found (1) substitution took place on a secondary carbon atom rather than a primary; (2) a second chlorine atom tended to substitute as far from the first chlorine atom as possible; (3) substitution of a second chlorine atom on a carbon atom already holding a halogen substituent was difficult—a third chlorine atom could not be introduced; (4) a bromine substituent was not displaced. In the chlorination of toluene the authors found also that the substitution of chlorine on a carbon atom ceased when the second chlorine atom had been introduced. The chlorination of ethylbenzenes and isopropylbenzene occurred chiefly in the alpha position of the side-chain. They were unable to chlorinate diphenylmethane and the nitrotoluenes presumably because of inhibition of the reaction by products of the reaction in the first case, and because of inhibition by the nitro-group in the second case. Complete inhibition was found to be brought about by a number of substances, iodine, sulphur, *p*-nitrotoluene, and oxygen among them. The mechanism of the reaction is considered to be the production of chlorine atoms by a chain reaction initiated by the slow decomposition of the peroxide.
R. D. S.

309. Nitrogen Compounds in Petroleum Bases. XV. Counter-Current Extraction of Kero Bases. Isolation of 2 : 4-Dimethyl-8-*n*-Propylquinoline. W. N. Axe and J. R. Bailey. *J. Amer. chem. Soc.*, 1939, **61**, 2609-2612.—A $C_{14}H_{17}N$ base was isolated by fractional degassing of the acid sulphite preliminary to exhaustive counter-current hydrochloric acid extraction of an aromatic kerosine fraction 292-293°. It was identified as 2 : 4-dimethyl-8-*n*-propylquinoline by (1) chromic acid oxidation to 2 : 4-dimethylquinoline-8-dicarboxylic acid, which was identical with an authentic specimen, (2) synthesis from acetylacetone and *o*-*n*-propylaniline.
R. D. S.

310. Nitrogen Compounds in Petroleum Distillates. XVI. Use of Multiple Acid Extraction of 2 : 3 : 4-Trimethyl-8-Ethylquinoline. R. A. Glenn and J. R. Bailey. *J. Amer. chem. Soc.*, 1939, **61**, 2612-2613.—Multiple acid extraction of a 305-315° kerosine fraction was used in the isolation of a $C_{14}H_{17}N$ base, the structure of which was identified as 2 : 3 : 4-trimethyl-8-ethylquinoline by chromic acid oxidation to a $C_{12}H_{12}NCOOH$ acid which proved replacement of an ethyl radical by a carboxyl; the 8-position of the ethyl group was shown by the fact that the acid was identical with one previously obtained by chromic acid oxidation of 2 : 3 : 4 : 8-tetramethylquinoline. The structure was confirmed by synthesis from methylacetylacetone and *o*-ethylaniline.
R. D. S.

311. Nitrogen Compounds in Petroleum Distillates. XVII. Use of Multiple Acid Extraction in Isolation of 2 : 3 : 4-Trimethyl-8-*n*-Propylquinoline. L. M. Schenck and J. R. Bailey. *J. Amer. chem. Soc.*, 1939, **61**, 2613-2615.—By multiple acid extraction a $C_{14}H_{19}N$ base, b. p. 330°, was isolated from transformer oil and kerosine and identified as 2 : 3 : 4-trimethyl-8-*n*-propylquinoline. Orientation was established by chromic acid oxidation to the same acid as that produced by oxidation of 2 : 3 : 4-trimethyl-8-ethylquinoline reported in the previous communication. The nature of the propyl group was proved by synthesis from methylacetylacetone and *o*-*n*-propylaniline.
R. D. S.

312. Peroxide Effect in the Addition of Reagents to Unsaturated Substances. XXII. Addition of Hydrogen Bromide to Trimethylethylene, Styrene, Crotonic Acid, and Ethyl Crotonate. C. Walling, M. S. Kharasch, and F. R. Mayo. *J. Amer. chem. Soc.*, 1939, **61**, 2693-2696.—In the absence of oxygen or peroxides, trimethylethylene adds hydrogen bromide to give *tert.*-amyl bromide, but peroxidic catalysis produces some 64% of 2-methyl-3-bromobutane. The addition of product of hydrogen bromide to styrene is changed from α -phenylethyl bromide with no catalyst to 80% β -phenylethyl bromide with catalyst in solution. However, presence or absence of peroxide always results in the same product of addition of hydrogen bromide to crotonic acid, nor do peroxides catalyse the reaction with ethyl crotonate even in the light. In each case the normal β -bromo-derivative is formed. The authors discuss the various possibilities for this.

R. D. S.

313. Hydrogen Fluoride as a Condensing Agent. VIII. The Alkylation of Benzene by Esters. J. H. Simons, S. Archer, and D. I. Randall. *J. Amer. chem. Soc.*, 1939, **61**, 1821-2.—Reactions carried out in a copper bomb at 80-100° showed that in presence of hydrogen fluoride esters react with benzene to produce mainly alkylbenzenes. It was believed that as acids can acylate aromatic compounds in the presence of hydrogen fluoride, ketones would be present in the reaction products, since the liberated acid would react with either benzene or the alkylbenzene present; and expectations were realized in the isolation of ketones in sufficient quantities to permit identification in a number of experiments. Thus, acetophenone was identified from those reactions in which acetates were used. An explanation of the mechanism is proffered, based on ionic processes and supported by experimental results, which indicate it to be a more likely one than an olefinic mechanism previously put forward (McKenna and Sowa, *J. Amer. chem. Soc.*, 1937, **59**, 1204-1205; *J. Inst. Petrol. Tech.*, 1937, **23**, Abstr. No. 1230). *tert.*-Butylbenzene, *isopropylbenzene* and diphenylmethane were formed from benzene and the acetates. *n*-Butyl acetate and *sec.*-butyl-*isobutyrate* both gave *sec.*-butylbenzene.

R. D. S.

314. Reaction of Propene with *iso*Olefins in the Presence of Sulphuric Acid. V. N. Ipatieff, H. Pines, and B. S. Friedman. *J. Amer. chem. Soc.*, 1939, **61**, 1825-1826.—Although propene alone was not responsive to polymerization by 96% sulphuric acid, when mixed with more reactive olefins such as (i) *isobutene*, or (ii) *isopentenes*, and then contacted with the acid, it yielded, among other products, (i) 8-35% *heptenes*, and (ii) 16% *octenes* and 17% *tridecenes*, respectively, the percentages varying according to the conditions. The *heptenes* were then hydrogenated to mostly 2:2-dimethylpentane, 2:3-dimethylpentane, and some 2:2:3-trimethylbutane, which *heptanes* were identified by physical constants and Raman spectra analysis. With straight-chain olefins such as *n*-butene scarcely any of the propene reacted to form liquid hydrocarbons when contacted with sulphuric acid.

R. D. S.

315. Dehydration of *trans*-2-MethylcycloHexanol. C. Price. *J. Amer. chem. Soc.*, 1939, **61**, 1847-1849.—By the dehydration of *trans*-2-methylcyclohexanol with phosphoric anhydride a mixture of 1-methyl-1-cyclohexene and 3-methyl-1-cyclohexene was formed, which on hydrogenation gave a methylcyclohexane no different from authentic samples of this hydrocarbon. This investigation, therefore, could not confirm a report (Vogel, *J. chem. Soc.*, 1938, 1323-1333; *J. Inst. Petr. Tech.*, 1938, **24**, Abstr. No. 1429) that the methylcyclohexane formed by the above method was of different physical properties from the genuine hydrocarbon.

R. D. S.

316. Heats of Organic Reactions. VIII. Some Further Hydrogenations, including those of some Acetylenes. J. B. Conn, G. B. Kistiakowsky, and E. A. Smith. *J. Amer. chem. Soc.*, 1939, **61**, 1868-1876.—This paper is the last of the series, and the discussion includes a survey of the results of previous communications on this subject. Heats of hydrogenation of cyclic hydrocarbons, cyclic ketones, and acetylenic compound have been measured. The rather large variation in the *cyclo*-olefin series is claimed to be due to interactions of the newly formed bonds with non-bonded atoms and atomic groups in the proximity. As the cyclic ketones show a trend similar in

manner to that of the *cyclo*-olefins, and the acetylenes, likewise, are similar to the ethylenes in the variation of the heats of hydrogenation with substitution of the alkyl groups, further evidence is obtained that the variation in the heats of addition reactions are principally caused by the steric effects of substituents on the addenda used in the reactions. In an attempt to evaluate the character and magnitude of the effects, the authors, from observations on ethane, come to the conclusion that the positions of minimum energy are those in which the C-H bonds are farther apart—that is to say, the configuration is staggered. The greater stability of this structure is ascribed to the existence of repulsive interactions—steric hindrance—between non-bonded atoms in the polyatomic molecules, and these repulsive interactions result in inconstancy of bond energies. The relative magnitude of steric hindrance is assumed to be substantially affected only by substitutions on the same atoms to which the bonds considered belong. The effect of hindrance of internal rotation is believed to be due to steric hindrance and not to an independent mechanism. Accordingly, the authors emphasize the importance of steric hindrance in regard to the effects of substituents upon "standard" properties of functional groups in polyatomic molecules, regarding it as next to resonance in this respect.

R. D. S.

317. Internal Rotation and Resonance in Hydrocarbons. E. Gorin, J. Walter, and H. Eyring. *J. Amer. chem. Soc.*, 1939, **61**, 1876-1886.—The restricted rotation of the methyl groups about the C-C bond in ethane is due, according to recent experimental evidence, to the existence of a potential barrier of the order of 3000 cal., but the repulsion due to the hydrogen atoms cannot account for this. The authors calculate what the barrier height would be if the hydrogen atoms were replaced by protons, and discuss the lack of cylindricality of the C-C bond due to the presence of the methyl hydrogen atoms. This effect adds to the stability of the opposed configuration, and allowing for the effect of resonance involving double bond structures (the opposed structure can be reasonably considered stable), the required barrier height is obtained. The authors also make use of the effect of resonance with double-bonded structures to explain the alteration in physical properties, and conclude with a discussion on the evidence available by X-ray, infra-red, and Raman spectra studies concerning the relative stabilities of staggered and opposed configurations for long-chain and cyclic compounds.

R. D. S.

318. Validity of the Structure Assigned to *cyclo*-Octatetrene: Pyrolysis of *bis*-Quaternary Ammonium Hydroxides Related to 1:2- and 2:3-Butenes. C. D. Hurd and L. R. Drake. *J. Amer. chem. Soc.*, 1939, **61**, 1943-1945.—The structure of 1:3:5:7-*cyclo*-octatetrene was formulated by Willstätter and Waser as a result of experiments on the pyrolysis of the *bis*-quaternary ammonium hydroxide. The argument of the present authors is that conjugated double bonds need not necessarily have been produced exclusively in the reaction; for pyrolysis of 1:2-butane-*bis*-(trimethylammonium) hydroxide gave rise to ethylacetylene and methylallene but no 1:3-butadiene, and pyrolysis of 2:3-butane-*bis*-(trimethylammonium) hydroxide gave 1:3-butadiene and methylallene or (methylallene + dimethylacetylene), the latter being slightly in excess of the 1:3-butadiene. That 1:3-butadiene was not the sole product from the second reaction (as would be required by conjugated unsaturation in *cyclo*-octatetrene) leads the authors seriously to doubt the Willstätter structure.

R. D. S.

319. Reaction of Olefins with Solid Cuprous Halides. E. R. Gilliland, J. E. Seebold, J. R. FitzHugh, and P. S. Morgan. *J. Amer. chem. Soc.*, 1939, **61**, 1960-1962.—Ethylene, propylene, and *isobutylene* were found to be absorbed by solid cuprous chloride, the first-named also being absorbed by solid cuprous bromide, from which the authors infer that the reaction is typical of olefins and the cuprous halides. The olefins were absorbed by the halides mole for mole, except in the case of *isobutylene*, where complete saturation was not obtained, as, presumably, equilibrium was not reached. It was found that olefins may be recovered from gaseous or liquid mixtures by absorption with cuprous halides, and the authors give a method based on the fugacity data of Lewis, by which the theoretical maximum recovery may be calculated.

R. D. S.

320. Effect of Substitution on the Dissociation of Hexa-arylethanes. VII. meta- and para-Phenyl Groups. C. S. Marvel, M. B. Mueller, and E. Ginsberg. *J. Amer. chem. Soc.*, 1939, **61**, 2008-2010.—The authors prepared the tetraphenyl-di-*m*-biphenyl-ethane, and found by measurement of its magnetic susceptibility that it is dissociated to the extent of 11-12% in a 3.6% solution of the ethane in benzene at 25°, and in the same way found that the *p*-biphenyl derivative exists to the extent of 13-14% as the free radical under the same conditions. They concluded that the number of resonance forms of the free radical must play a rather minor rôle in determining the degree of dissociation of these hexa-arylethanes. They found, further, that the depth of colour of a free radical solution is not a reliable guide to the degree of dissociation, being merely partly characteristic of the groups present and their positions. R. D. S.

321. Synthesis of tert.-Butyl- and tert.-Amyl-cyclopentane and of their Intermediate Products. H. Pines and V. N. Ipatieff. *J. Amer. chem. Soc.*, 1939, **61**, 2728-2730.—The method consists of cyclizing the appropriate alkyladipic acid to alkylcyclopentanone, which is then hydrogenated catalytically to 3-*tert*.-alkylcyclopentanol. The carbinols are next dehydrated by the use of aluminium oxide to the olefins, which on hydrogenation give *tert*.-butyl- and *tert*.-amyl-cyclopentane. R. D. S.

322. The Redistribution Reaction. I. The Random Intermolecular Exchange of Organic Radicals. G. Calingaert and H. A. Beatty. *J. Amer. chem. Soc.*, 1939, **61**, 2748-2754.—A new type of reaction, which the authors propose to call "The Redistribution Reaction," has been recognized. The process is one in which all the radicals or atoms of one kind, linked to one or more central groups or atoms of another kind, are interchanged (during a reaction occurring in a mixture of such compounds) between all the various central groups present. As the final composition of any system of this kind can be calculated from the laws of probability, the interchange must happen at random, hence the term "Random Equilibrium Mixture" applied by the authors to the reaction products. Although this introductory paper considers the exchange of radicals in organo-metallic halides and in metal alkyls, the authors stress the fact that the redistribution reaction is not confined to this field. The first reaction studied was that between tetraethyl-lead and triethyl-lead chloride, the tetraethyl-lead containing radioactive lead (Radium D). After a short time at room temperature both the tetraethyl-lead and the triethyl-lead chloride showed the same intensity of radio-activity. A repetition of the experiment was performed with the alkyl group in the alkyl-lead different from that in the alkyl-lead halide; here all the five possible tetra-alkyl-leads and all the four possible trialkyl-lead chlorides were formed. The reactions between the two different metal alkyls were studied. The authors found that, employing various catalysts, the reaction proceeded smoothly between the compounds containing the same or different alkyl groups and the same or different metals, with no decomposition, side reactions, evolution of gas, precipitation of metal, or heat of reaction. The system was found to reach a state of constant composition, beyond which it could not proceed and containing all the possible metal alkyl compounds in equilibrium. The authors have applied the laws of probability to these random equilibrium mixtures and have confirmed their findings experimentally. They consider the mechanism of the redistribution reaction to proceed through the formation of co-ordination compounds with the catalyst, and observe that the reaction has possibilities for investigations in the nature and behaviour of covalent bonds as well as for organic synthesis. R. D. S.

323. The Redistribution Reaction. II. The Analysis of Metal Alkyl Mixtures and the Confirmation of Random Distribution. G. Calingaert, H. A. Beatty, and H. R. Neal. *J. Amer. chem. Soc.*, 1939, **61**, 2755-2758.—This paper describes how the analyses of the volatile metal alkyl mixtures were carried out by distillation correct to less than 1%, and how the analyses of a series of mixtures which had suffered the redistribution reaction agreed with the predicted compositions. R. D. S.

324. The Redistribution Reaction. III. Determination of a Material Balance. G. Calingaert and H. Soroos. *J. Amer. chem. Soc.*, 1939, **61**, 2758-2760.—The redistri-

bution reaction was shown to be quantitative in nature by the determination of an accurate material balance for a reaction involving a mixture of tetramethyl- and tetraethyl-lead in hexane solution. It was found that the use of aluminium chloride as catalyst resulted in the formation of only very small quantities of alkyl-lead compounds, whilst with methyl aluminium chlorides the amount of alkyl-lead compounds formed was negligible.

R. D. S.

325. Effect of Substitution on the Dissociation of Hexa-arylethanes. VIII. The Disproportionation of Tri-*p*-Tolymethyl. C. S. Marvel, W. H. Rieger, and M. B. Mueller. *J. Amer. chem. Soc.*, 1939, **61**, 2769-2771.—Magnetic susceptibility measurements showed that hexa-*p*-tolymethane in fresh benzene solution is dissociated to the extent of $16 \pm 2\%$ in 0.08 molar concentration at 30°, although if the solution is allowed to stand for a few hours at room temperature the colour disappears, and a new determination shows that the free radical is no longer present, but a polymeric material remains. The authors suggest a mechanism for the disproportionation based on that of tri-*p*-tolymethyl, which, in benzene solution, rapidly forms tri-*p*-tolymethane and a quinoid polymeric residue. The same polymer seemed to be produced by the removal of hydrogen chloride from tri-*p*-tolymethylchloride by the action of pyridine.

R. D. S.

326. The Disproportionation of Hexa-*p*-Alkylphenylethanes and the Effect of *ortho*-, *meta*-, and *para*-Alkyl Groups on Dissociation of Hexa-arylethanes. C. S. Marvel, M. B. Mueller, C. M. Himel, and J. F. Kaplan. *J. Amer. chem. Soc.*, 1939, **61**, 2771-2775.—In the ninth communication on the effect of alkyl groups on the dissociation of hexa-arylethanes the authors report that those hexa-*p*-alkylphenylethanes in which there is a hydrogen atom on the α -carbon atom of the alkyl group dissociate to free radicals which undergo disproportionation. They found that the effect of the various *p*-alkyl groups on the degree of dissociation (17-33%) was greater than expected (2-6%), and varied according to the shape and size of the alkyl group. They also found that the *ortho*- and *meta*-methyl groups, due partly to steric effects, were more active than a *para*-methyl group in promoting dissociation and gave relatively stable radicals. Di-*p*-alkylphenyltetraphenylethanes were found to be more highly dissociated than hexaphenylethane and to disproportionate less readily than the corresponding hexa-alkyl derivatives.

R. D. S.

327. Hydrogenation of Substituted Acetylenes with Raney Nickel. K. N. Campbell and M. J. O'Connor. *J. Amer. chem. Soc.*, 1939, **61**, 2897-2900.—Using Raney nickel as a catalyst, the authors have reduced mono- and di-substituted acetylenes, the course of the reaction being shown to depend on the symmetry of the molecule. At the half-reduction stage certain new olefins were obtained from some of the dialkylacetylenes, and the authors suggest that the use of Raney nickel, which seems to act catalytically like palladium, but is easier to use, affords a convenient method for making olefins by half reduction and saturated hydrocarbons by full reduction.

R. D. S.

328. Fluorocarbons. The Reaction of Fluorine with Carbon. J. H. Simons and L. P. Block. *J. Amer. chem. Soc.*, 1939, **61**, 2962-2966.—The properties of six compounds in addition to a 25-160° boiling-range mixture prepared by a continuous non-explosive reaction between fluorine and carbon are described.

R. D. S.

329. Benzoylformic Acid from Styrene. C. D. Hurd, R. W. McNamee, and F. O. Green. *J. Amer. chem. Soc.*, 1939, **61**, 2979-2980.—An account is given of the oxidation of styrene (phenylethylene) to benzoylformic acid by potassium permanganate.

R. D. S.

330.* Phase Equilibria in Hydrocarbon Systems: The Methane-Ethane System in the Gaseous Region. B. H. Sage and W. N. Lacey. *Industr. Engng Chem.*, 1939, **31** (12), 1497.—The specific volumes of five mixtures of methane and ethane were determined at 70, 100, 130, 160, 190, 220, and 250° F., and pressures up to 4750 lb. per sq. in. The results are presented in tabular form. The partial volumetric behaviour of

methane and ethane in the system has been calculated, and from these data and published information the partial thermodynamic behaviour of the components has been calculated over the range of conditions given above, and presented in graphical and tabular form.

P. D.

331.* Halogenation of Hydrocarbons. H. P. A. Groll and G. Hearne. *Industr. Engng Chem.*, 1939, **31** (12), 1530.—Olefines containing a double bond in an unbranched carbon chain react with halogens at elevated operating temperatures to give a product which is almost exclusively of the substitution type. The substitution occurs in the olefine with the almost exclusive formation of allyl-type unsaturated monohalides. The optimum temperature range for this reaction is 300–600° C., and depends on the nature of the reacting olefine and halogen. Operating conditions and yields have been carefully examined in some detail, and although at present the exact mechanism of the reaction is obscure, the data presented will prove useful as a basis for the study of the kinetics of the reaction. The main section of this valuable paper is concerned with the chlorination of propylene at elevated temperatures, and the various factors affecting the reaction have been investigated in considerable detail. The corresponding bromination of propylene has only been attempted for a few isolated cases.

H. E. T.

332.* Viscosity Index.—Thomas. *Proc. II^{me} Congrès mond. Pétrole*, 1938, **2** (2), 891–896.—Several graphical methods have been proposed for expressing the change in viscosity of an oil with temperature. A number of these graphs are reproduced and discussed. The relation of viscosity–temperature characteristics to cold starting, oil consumption, etc., is demonstrated. Suitable increase in viscosity index should enable the same oil to be used throughout the year.

W. P.

333.* Viscosity Index Chart for Kinematic Viscosities. F. H. Garner and E. W. Hardeman. *Proc. II^{me} Congrès mond. Pétrole*, 1938, **2** (2), 897–899.—Absolute units are now more generally used for expressing viscosity of lubricating oils. Using the Dean and Davis formula for viscosity index, the authors have constructed a viscosity index chart for kinematic viscosities using viscosities determined at 50° C. and 100° C.

W. P.

334.* Graphs for Determination of Viscosity Index. M. Roegiers. *Proc. II^{me} Congrès mond. Pétrole*, 1937, **2** (2), 901–904.—The Dean and Davis method of deriving viscosity-index based on Saybolt viscosities at 210° F. and 100° F. is limited in its application to America, since neither the Saybolt nor the particular reference temperatures are normally used in Europe. Graphs are given which enable the viscosity index to be determined from viscosities at 20° C. and 100° C. (Continental) or 70° F. and 100° F. (English).

W. P.

335.* Viscosity Index Independent of Temperature of Measurement. F. P. Malschaert. *Proc. II^{me} Congrès mond. Pétrole*, 1938, **2** (2), 905–910.—Viscosity–temperature characteristics have been represented by two systems: (a) in which log log (centistokes + 0.8) is plotted against log *T*, e.g. the Viscosity Pole-height system, (b) in which the viscosities at two temperatures are related to viscosities of standard oils, e.g., Dean and Davis system. The advantages and disadvantages of the two systems are discussed.

The author proposes an Independent Viscosity Index, I.V.I. = $100 \times \frac{L - X}{L - H}$, where *L*, *H*, and *X* are log log (centistokes + 0.8), relating respectively to 0KVI, 100KVI and unknown oils.

The advantages claimed are: (1) ease of plotting, (2) extrapolation beyond 0 and 100, (3) application to mixtures, (4) change of base oils possible, (5) similarity of I.V.I. and K.V.I. within region 0–100.

W. P.

336.* Nomograms for Expression of Viscosity of Lubricating Oils. J. Groff. *Proc. II^{me} Congrès mond. Pétrole*, 1938, **2** (2), 911–915.—The logarithmic graphs derived

from the A.S.T.M. formula have been converted into nomograms with scales in various conventional units. W. P.

337.* Viscosity-Temperature Curve of Lubricating Oils. L. Steiner. *Proc. IIme Congrès mond. Pétrole*, 1938, 2 (2), 925-931.—Viscosity curves have been prepared for a number of oils from determinations of viscosity with a Steiner viscometer between 20° C. and 100° C. Results were examined for conformity with the formula $\log \log (V + a) = A - B \log T$. It is concluded:—

(1) That the formula holds sufficiently accurately between 20° C. and 100° C. for normal practical purposes, and that the divergence between actual and theoretical is the same for all oils.

(2) That the value of a is not constant, but lies between 0.6 and 1.2, with an average of 0.8.

(3) That if the fixed points are accurately calculated, extrapolated values are lower, and interpolated values higher than actual with an average error of $\pm 5\%$. W. P.

338.* Viscosity-Temperature Relationships of Hydrocarbons. E. B. Evans. *Proc. IIme Congrès mond. Pétrole*, 1938, 2 (2), 933-954.—The viscosities of ninety-three hydrocarbons are given in centipoises at 0°, 20°, 50°, 80°, and 100° C. These data are applied to a discussion of various viscosity-temperature relationships, and it is concluded that the A.S.T.M. equation, a modified Andrade-Silverman equation, and a modified Batschinski equation are of outstanding interest, since they are capable of representing accurately a wide range of data and there is considerable theoretical justification for the last two. W. P.

339.* Viscosity of Pure Hydrocarbons. W. R. Van Wijk and J. M. Versteeg. *Proc. IIme Congrès mond. Pétrole*, 1938, 2 (2), 955-959.—The viscosity of a number of pure hydrocarbons has been determined with a view to studying the viscosity-temperature and viscosity-density characteristics in relation to structure. Normal hydrocarbons from C_5 to C_{14} have approximately equal viscosities at equal densities (within 5%). In general the viscosity of branched-chain hydrocarbons is greater than that of the normal series, but in some cases the reverse is true. For normal series the viscosity, considered as a function of density, appears to pass through a minimum in the neighbourhood of C_9 . W. P.

340.* Logarithmic Relation Between Viscosity and Temperature. G. Juge-Boirard. *Proc. IIme Congrès mond. Pétrole*, 1938, 2 (2), 961-971.—Viscosity-temperature formulae from Poiseuille to Walther are criticized as attempting to introduce a constant or constants of universal application. The author deduces a simple linear logarithmic relation between viscosity and temperature

$$\log \eta t = a \log \frac{t + c}{t_1 + c} + \log \eta_1$$

in which c is a constant particular to the material in question and calculable from viscosity data determined at three fixed temperatures.

The equation can be adapted to any temperature scale. W. P.

341.* Viscosity-Temperature Relationship. E. L. Lederer. *Proc. IIme Congrès mond. Pétrole*, 1938, 2 (2), 973-979.—The "Steilheitsgefälle N" is defined as the second differential of viscosity with respect to temperature, and a formula is derived relating it to absolute temperature. The thermodynamical basis of the formula and its relation to chemical constitution are discussed in comparison with other systems, such as that of "Pole-height." W. P.

342.* Constitution in Relation to Viscosity of Mixtures. A. Bondi. *Proc. IIme Congrès mond. Pétrole*, 1938, 2 (2), 987-989.—A consideration of the theoretical consequences

of the Arrhenius formula for the viscosity of mixtures leads to the evaluation of the magnitude and temperature susceptibility of the "Solvation factor" in the Lederer formula. Their relation to chemical constitution is shown by viscosity measurements on a number of oils and oil mixtures. W. P.

343. Catalytic Dehydrogenation of Mono-Olefines to Diolefines. A. V. Grosse, J. C. Morrell, and J. M. Mavity. *Symposium on Plastics and Resins from Hydrocarbons. Amer. chem. Soc. Meeting, Boston, September 1939, pp. 53-61.*—A previous paper has described the catalytic dehydrogenation of gaseous paraffins to mono-olefines. The same process using the same catalysts can be applied for the dehydrogenation of olefines to diolefines when products of the conjugated type with the same C framework as the parent olefines are formed.

Olefines the C frameworks of which do not permit the introduction of conjugated double bonds yield under these conditions hydrogen and carbonaceous material.

In these experiments the charge was passed at a definite rate through a section of granular catalyst in a quartz tube maintained at a constant controlled temperature. The catalysts in general were of the chromium, molybdenum, or vanadium oxide-on-alumina type. The diolefines formed were identified by the preparation of crystalline derivatives, either the maleic anhydride addition compounds, the tetrabromides, or both.

The once-through yields of the diolefines under the conditions used in these experiments varied from 20% to 30%, but it is possible that these do not represent optimum conditions.

Carbon formation in the dehydrogenation of olefines is considerably higher than in the dehydrogenation of paraffins.

The particular diolefines prepared in this work were butadiene-1:3 from *n*-butylenes, isoprene from branched-chain pentenes, and piperylene from pentene-2. It is pointed out that the production of cheap butadiene and isoprene is of great importance in the development of the synthetic rubber industry. D. L. S.

344. Action of Boron Halides on Hydrocarbons. R. F. Ruthruff. *Symposium on Plastics and Resins from Hydrocarbons. Amer. chem. Soc. Meeting, Boston, September 1939, pp. 63-91.*—Considerable attention has been given to reactions catalysed by aluminium chloride, but analogous work employing the boron halides has been comparatively meagre.

Boron halides are usually more active than the aluminium halides, and the reactions induced are more clean-cut and less masked by side reactions. The preparation and properties of the four halides are described, boron fluoride being dealt with in more detail than the other three.

The greater part of the paper then discusses the action of boron fluoride on olefinic hydrocarbons, a short paragraph only dealing with the effect of the other three halides.

Boron fluoride has little action on the paraffin hydrocarbons alone, although in the presence of olefines some of these substances are alkylated. The effect of this catalyst on the polymerization of olefines is described in some detail and the work of numerous investigators is discussed.

Conditions for polymerizing ethylene, propylene, butylenes, amylenes, and many other olefines are given. The low-temperature polymerization of *isobutylene* and other *iso*-olefines yields products of a resinous or rubber-like type which find numerous applications in industry.

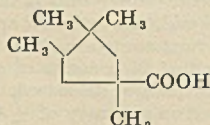
In general the activity of the catalyst is improved by the use of a promoter, and finely divided nickel, hydrogen fluoride, hydrogen chloride, and organic halides are amongst the materials used for this purpose.

An attempt to deal with the olefine-boron fluoride complexes formed in these reactions from the standpoint of electronic structure is made. D. L. S.

345. Solid Naphthenic Acid from Iranian Petroleum. T. Kennedy. *Nature*, 11th November, 1939, 144, 832.—It is only comparatively recently that individual naphthenic acids have been isolated in a pure state, many which had previously been reported as pure being subsequently proved to be mixtures.

Examination of the SO₂ extract from an Iranian kerosine revealed unexpectedly

the presence of approximately 0.025% of naphthenic acids, of which about 10% was removed by a light caustic washing. The spent caustic washings from 2000 gals. extract were neutralized and the precipitated oil was worked up in the usual manner. From the acidic material the tertiary acids were separated, and these, on standing for a few days, yielded a white crystalline solid which analysis showed to have an empirical formula $C_{10}H_{18}O_2$. A total yield of 3.3% of this acid was obtained on the original acid mixture from the caustic washing-liquors. The compound was proved to be saturated, and must therefore contain a closed carbon ring, the following structure being suggested



Since this work was completed the isolation of a solid tertiary naphthenic acid, m. p. 194–194.5° C., from a Californian straight-run gasoline distillate has been described by other workers.
D. L. S.

346.* **New Catalytic Syntheses of Hydrocarbons.** V. N. Ipatieff. *Monit. Pétr. roum.*, 1939, 40, 1433–1438.—Recent developments in alkylation, cyclization, and polymerization are reviewed. The elements of the structural changes during these syntheses are presented.
T. C. G. T.

Analysis and Testing.

347.* **Means of Measuring Turbidity and Fluorescence Using the Lovibond Tintometer Fitted with the Rothamsted Device.** G. S. Fawcett and J. Hewitt. *J. Soc. chem. Ind.*, 1939, 58, 342–344.—A special cell, termed a nephelometer cell, has been developed for the Lovibond-Schofield apparatus, which latter is a modification of the Lovibond tintometer. Using the nephelometer cell, turbidities of colourless liquids can be measured easily in quantities ranging from 0 to 100 parts of fuller's earth per 100,000 parts of water.

The cell is placed at the back of the artificial light cabinet, and a piece of polished black glass is placed behind and in contact with the cell. Light from the lamps passes through the cell at 45°, and the light scattered by the particles in the liquid illuminates one field of the eye-piece. The other field is illuminated by light reflected from a magnesium carbonate block, and the brightness of the field is adjusted until it matches that of the other. The brightness of varying degree is obtained by Schofield's rotating vane.

The cell may also be utilized for the measurement of fluorescence. In this case the fluorescent liquid is diluted to remove if possible the predominating colour of the liquid, but to retain the fluorescence. The fluorescent colour is matched by colour-slides.
T. C. G. T.

348.* **Laboratory Method for Evaluating the Influence of Lubricating Oils on Carbon Deposition.** H. A. Everett and G. H. Kaller. *Engineering*, 1939, 148, 676.—Two types of small, single-cylinder, air-cooled engines were used to evaluate the carbon-forming tendencies of different lubricating oils. In order to eliminate the inevitable variables of slight differences in engine performance from run to run, and to eliminate the effect of progressive wear during a series of tests, six engines of each type were used, and the tests carried out so that each engine ran alternately on the reference oil and the test oil. The various oils were tested in each of the six units of both types of engine. The carbon built up on the cylinder heads was determined at intervals of 8, 16, 24, 32, and 40 hr. of operation by weighing the head and replacing without removing the carbon. At the end of 40 hr. the pistons were weighed to determine the build-up on the piston. The total oil consumption was also measured and the carbon deposited in the combustion space expressed as the *specific carbon* (weight of carbon deposited divided by weight of oil consumed). The *specific carbon value* for an oil divided by the *specific carbon* of the reference oil gives the *carbon ratio*. It is

shown that for the five oils examined the carbon ratios as determined in either type of test engine were in good agreement with the carbon ratios obtained on full-sized six-cylinder automobile engines.

J. W. H.

349.* Errors in the Calibration of Secondary Viscometers. A. Gangloff. *Proc. II^{me} Congrès mond. Pétrole*, 1938, 2 (2), 887-889.—A description of improvements in the methods and apparatus used for the calibration of absolute viscometers. The calibration involves determination of constants h and K in the expression $\eta = K(H - ha)t$; h being back pressure due to liquid, H suction, a specific gravity, and t time of flow. For the determination of h two runs are made, one with suction H , one under action of gravity. For the determination of K a series of standard reference viscometers is used. Errors of earlier methods are discussed, and thermostatically controlled apparatus is described capable of an accuracy of $\pm 0.005^\circ \text{C}$.

W. P.

350.* Flow of Oil at Low Temperatures. S. Erk. *Proc. II^{me} Congrès mond. Pétrole*, 1938, 2 (2), 917-923.—The ability of an oil to flow at low temperatures is important from the point of view both of cold starting and of the maintenance of adequate lubrication. Conventional methods of testing for flow at low temperatures are criticized, in that they do not take into account all the practical requirements. Microscopic investigation shows that the "cold point" coincides with the commencement of crystallization of wax and that flow under mechanical forces is due to the break-up of the crystal skeleton. In this condition the oil flows, but its viscosity is dependent on the rate of shear. True evaluation of an oil at low temperatures necessitates determination of flow and viscosity in relation both to temperature and to rate of shear.

W. P.

351.* Apparatus for Testing Lubricants and Bearing Metals. J. Prevost. *Proc. II^{me} Congrès mond. Pétrole*, 1938, 2 (2), 1011-1016.—The Vollet machine consists essentially of a free-running ring inserted between two discs which are fixed to concentric shafts whereby they can be rotated. The lubricant under test is fed on to the ring which tends to be entrained by the rotating discs, and the couple exerted can be determined.

A mathematical analysis of the machine is given and a number of results quoted. The machine can be used for tests on lubricants or on bearing metals.

W. P.

352.* Nomograms for Expressing Results of Mechanical Tests on Lubricants and Bearing Metals. J. Prevost. *Proc. II^{me} Congrès mond. Pétrole*, 1938, 2 (2), 1017-1020.—Mechanical tests of lubricants generally permit only comparative results. The interpretation of such results can be facilitated by graphical presentation, and a nomogram is given based on results achieved with the Vollet machine, but of a type applicable to other tests.

W. P.

Motor Fuels.

353.* Development of Anti-knock Motor Fuel Part 2. A. W. Trusty. *Petrol. Engr*, October 1939, 11 (1), 34-36.—Two general methods are available for improving the anti-knock qualities of motor fuel: (1) addition of a chemical, (2) changing the hydrocarbon structure of a gasoline. Of chemicals added, tetraethyl lead is the most widely used. Safety regulations to ensure that the handling of this poisonous compound is not dangerous to the health of the operator are described. The inclusion of ethylene bromide in the fluid provides bromine to convert the lead into lead bromide which is sufficiently volatile to pass out of the cylinder with the exhaust gases. Iron and nickel carbonyls are effective as anti-knock dopes, but have not been commercially developed to the same extent as tetraethyl lead. The usefulness of benzol and alcohol as anti-knock compounds is illustrated by a tabulation of their properties in comparison with those of a typical gasoline. High anti-knock gasoline can also be produced by solvent extraction of selected fractions. Results obtained with sulphur dioxide are illustrated.

The maximum octane number gasoline that can be practicably produced by liquid

phase cracking is about 70. Higher anti-knock gasolines are obtained by developments of cracking, viz. reformation, alkylation, polymerization, and hydrogenation. Reformation is used to convert a low octane s.r. gasoline into a high octane fuel. Alkylation consists of the combination of an olefine with a paraffin in the presence of concentrated sulphuric acid to give a branch-chain paraffin. By segregation of suitable hydrocarbons, fuels with an octane number of 90-100 can be produced by this process. Paraffin dehydrogenation is effected by the use of alumina catalyst activated by metallic oxides and salts. Dehydrogenation of propane and butane followed by polymerization and hydrogenation gives high anti-knock *iso*-paraffins. Aromatic hydrocarbons can be formed by polymerization of olefinic gases, but, although high in anti-knock value, these hydrocarbons have the disadvantage of a poor lead response. The polymerization process has been operated either with phosphoric acid as catalyst at relatively low temperatures and pressures or at high temperatures and pressures without catalyst. Both methods yield a polymer gasoline with a blending value of about 120 octano number.

R. J. E.

354. Hydrogen as a Substitute Fuel for Otto Engines. M. Oelmichen. *Automobiltech. Z.*, 1939 (42), 573-576.—When an emergency arises in a country in which there is a shortage of liquid fuels, the possibility of the use of hydrogen as a substitute fuel for Otto engines is invariably reconsidered. During the last war and afterwards a great deal of research work was done in Germany by Erren, and a company formed to store the surplus energy of the big power-stations in the form of hydrogen formed by the electrolysis of water, and to use it in motor-cars and industrial power-stations. Difficulties in the use of hydrogen are to be found in the fact that it strongly attacks all iron, and especially valves, so that a sleeve-valve engine of special design has to be used. Moreover, hydrogen gives far too quick a combustion. It has therefore been proposed to mix it with inert gases before entering the cylinders. The author summarizes all available data on the subject, and concludes that the fundamental principles of the question still need elucidating.

E. W. S.

Diesel Fuels.

355.* Improving Diesel Fuels by Blending with Fischer-Tropsch Synthesis Products. H. Kölbel. *BrennstChemie*, 1939, 20, 352-355, 365-369.—Kogasin II produced by the Fischer-Tropsch synthesis would permit a considerable reduction of the compression ratio (to 1:11 or 1:10), but it is too expensive to be used alone, and its extremely short ignition delay would prohibit its use in present-day C.I. engines. Also, its high freezing point, combined with paraffin-wax crystal formation commencing at about 2° C., are a disadvantage, whilst dewaxing is not feasible. Suitable fuels can, however, be prepared when Kogasin II is blended with low-ignition-quality aromatic tar oils or hydrogenated oils. The percentage of Kogasin II added varies, according to the ignition quality of the poorer component, between 40% and 55%, and the fuel blends obtained have cetene numbers between 65 and 85. Blends with unrefined tar oils cannot be stored and are not suitable for engine use. For such oils special methods of refining have been developed. The chemical properties and engine behaviour of a series of fuel blends containing coal-tar oil, internal-suction tar oil, and coal-hydrogenation oil, respectively, which had been produced according to these methods, partly in large-scale experiments, and partly in large production plants, are examined in detail. Those blended fuels are thoroughly equal to petroleum products. They have a high ignition quality, a high resistance to carbon formation, and an extremely good combustion.

L. R.

356.* Production of Vegetable Fatty Oils in France—Soya-Bean Oil. A. B. d'Ollivier. *Rev. Comb. lig.* (August-September), 1939 (167), 225-235.—In a discussion on the cultivation of the soya bean in France, reference is made to the value of the oil as a diesel engine fuel. Experiments carried out in 1933 by the South Manchurian Railway at a temperature of -18° to -25° C. proved conclusively its suitability as fuel for heavy oil engines. It was particularly noted that starting was effected without any pre-heating.

The following table gives a comparative analysis between French and Manchurian soya-bean oil.

	French soya-bean oil.	Manchurian soya-bean oil.
Density at 15° C.	0.933	0.920
Acidity as oleic	0.22%	0.15%
Calorific value	9392	9340
Flash point	314° C	—
Viscosity Engler at		
10° C.	14.13	—
15° C.	—	6.1
20° C.	8.78	—
35° C.	—	4.5
50° C.	3.18	—
55° C.	—	2.7

Research work has been carried out at Clermont-Ferrand with a diesel motor, using soya and peanut oils as fuels, gas oil having a cetene value of 65 being used for comparison.

The motor was a four-stroke two-cylinder type, having a preheating chamber, a bore of 126 mm., a stroke of 180 mm., and working at 1140 r.p.m. The engine was not modified in any way, except by the provision of a small electric heater in order to reduce the viscosity of the oils under test by bringing them to a temperature between 55° and 60° C.

Comparative consumption tests were carried out at full, $\frac{2}{3}$, and $\frac{1}{3}$ loads, and a table is given showing the results, these being in all cases somewhat higher than for gas oil for both soya and peanut oils, the former showing the highest consumption in gm./h.p./hr.

Using the vegetable oils it was not found possible to attain the same h.p. as with gas oil, but this is explained by the fact that the injection pump was not set correctly, and consequently the oil delivery was insufficient. In view of the fact that no exhaust fumes were observed in the above tests, it is suggested that by increasing the oil delivery, the output attained with gas oil could have been reached. Exhaust gases analysis showed that an output of 32.2 h.p. was attained with the same mixture strength for the three fuels, this being about 0.65. Starting was found to be quite satisfactory for all three fuels, a torch of special paper being used as a hot spot. An examination of the engine after each test showed no abnormal deposit on the injector, valves, or any other parts of the engine.

These first tests have proved conclusively that the diesel motor under test gave a satisfactory performance using soya and peanut oils as fuels, but in view of the fact that the engine used is particularly suitable for use with a variety of fuels (relatively slow speeds and preheating chamber), and that the small quantity of fuels available was not sufficient for exhaustive tests, it is suggested that further work should be carried out on a greater variety of engines, particularly of the industrial types, before a thorough study of lorry motors is attempted.

M. M. L.

Lubricants and Lubrication.

357.* Properties of Mineral Lubricating Oils. — Freund. *Proc. II^eme Congrès mond. Pétrole*, 1938, 2 (2), 981-986.—In previous publications the author has shown certain relationships between viscosity, density, boiling point, molecular weight, etc. To investigate whether these same relationships held good for oils prepared by newer methods, e.g., solvent extraction, hydrogenation, etc., the author has examined a number of high-class commercial oils of low specific gravity manufactured by different processes. In general, the relationships formerly established were found still to hold good, except in the case of synthetic oils.

W. P.

358.* Improved Lubricants and Lubrication. B. H. Lincoln and G. D. Byrkit. *Proc. II^eme Congrès mond. Pétrole*, 1938, 2 (2), 991-1001.—The general theory of lubrication is discussed, and the results of X-ray and other investigations into film structure and film thickness are reviewed.

Super refining, although improving viscosity-temperature and other characteristics of oils, removes those polar bodies to which "oiliness" is due. Selection of suitable

additives can be made by preliminary laboratory investigation followed by service tests. The results of laboratory investigation of a number of additives using the Timken Wear Test and the Almen Wear Test are given. W. P.

359.* The Molecular Theory of Lubrication. O. Wittrock. *Proc. IIme Congrès mond. Pétrole*, 1938, 2 (2), 1003-1009.—Experimental confirmation of the hitherto purely theoretical hydro-dynamic theory of lubrication has been attempted. Assuming perfect metal surfaces, a formula can be readily deduced for liquid friction on this hypothesis. An apparatus is described for the experimental investigation of the formula. A film of lubricant is observed between two circular metal surfaces formed by the ends of two cylinders, one of which can be rotated. The pressure on the film can be regulated and the film thickness measured optically.

It is concluded that it is necessary to modify the formula to take into account the nature of the metal surfaces, and that in boundary friction the effect of the physical state of the metal surfaces is normally greatly in excess of that of the molecular structure of the oil. W. P.

360.* Used Lubricating Oil Recovery. A. T. Wilford. *Petroleum*, December 1939, 1 (2), 41.—A discussion is presented of methods of recovery of used lubricating oil, based on experience of the London Passenger Transport Board.

Of the lubricants used in motor vehicles only the engine oil and oil from pre-selective gear-boxes is regarded as suitable for recovery, that from clash gear-boxes and rear axles being too small in quantity and very much oxidized in service, whilst that from fluid flywheels is not worth recovery. It is estimated that at a consumption of 1 gal. per 1000 miles, the ratio of oil available for recovery to total used is 5 : 11 if crankcases are drained every 6000 miles, or 5 : 17 if drained every 12,000. Pre-selective gear-boxes provide a maximum of 1½ gal. after 6000 miles running which it is not always profitable to recover, as there are few purposes for which it could be used, except possibly as fuel. Continuous purification *in situ* using felt, cotton waste, or filter clay is frequently used on goods and passenger vehicles, the type using replaceable cartons being regarded as the most efficient, although much more expensive to maintain. The clay type are claimed to remove asphaltic and acidic bodies, but this shortens their useful life, and may lead to the absorption of any inhibitors present in the oil.

Crank-cases of petrol engines of the L.P.T.B. are drained every 12,000 miles and re-filled with a mixture of fresh oil and 10% of used oil. With high-speed oil-engines of the indirect injection type, oil is drained after 12,000 miles, and with those of the direct-injection type after 6000 miles; in neither case is the oil used again.

Figures are given showing typical characteristics of used oils from petrol and high-speed oil-engines and from pre-selective gear-boxes, indicating the much higher hard asphalt content of the oil from petrol engines than that from oil engines, owing to its being of a lower grade, whilst the carbon is in a less finely divided condition.

Methods of recovery of such oils are described. Centrifuging is used for lightly contaminated oils from large diesel engines and batteries of engine-test stands; coagulation of carbon in used oil from petrol engines is effected by mixing with 1-2% of an alkali, agitation and heating by steam, and separating the oil and centrifuging, followed by further mixing with water and centrifuging. Final heating of the oil removed the last traces of diluent and left the oil suitable for re-use. Owing to the formation of emulsions, oils from C.I. engines could not be satisfactorily treated by this method.

Edge filtration through paper discs removes suspended matter, but only partly reduces asphaltic or acidic compounds. Some improvement is effected by increasing the temperature of the oil and using filter aid. C.I. engine oil is difficult to reclaim by this method, but pre-selective gear-box oil is normally recovered by it and re-used once, although it is inferior in oxidation resistance to the original oil. Another type of filter, consisting of pairs of papers impregnated with kieselguhr crimped at the edges and separated at the centres by ¼-in. washers, is claimed to be suitable for C.I. engine oil.

Impurities from used petrol-engine oils can also be removed by floating it over a large surface of water containing a coagulant such as trisodium phosphate, which slowly rises through the oil through electrical heating. On cooling, the water settles, taking the impurities with it.

Mixing the heated oil with absorbent clays is capable of treating used C.I.-engine oil, yielding oils closely resembling the fresh oil.

Distillation followed by sulphuric-acid treatment, clay treatment, and filtration requires careful handling, and is suitable only where large quantities of oil are available. Yields of recovered oil vary from 80% to 95%, whilst the cost may approach 1s. per gallon. It is emphasized that spectacular economies cannot be expected, although small definite savings are being effected, and the present conditions suggest the extension of such work. It is suggested that the best and most economical method is for the oil to be re-run in refineries handling crude oil. C. L. G.

361.* Recent Developments in Diesel Lubricating Oils. G. L. Neely. *J. Soc. Aut. Engrs.*, 1939, 45 (5), 485-500.—The author contends that lubrication is an element of machine design; suitably compounded diesel lubricants are justified, inasmuch as they allow engine performance to be extended to a higher level than is possible with straight mineral oils. The paper describes the development of a new compounded diesel lubricating oil; the base stock used is a specially treated naphthenic-base oil. The necessary development work involved prolonged tests on five makes of engines using eight sets of test conditions; loads ranged from 2.5% to 150% of rated power and jacket temperatures from 80° F. to 375° F. The results given in the paper are confined to four compounded oils and one straight mineral oil.

The use of compounded oils led to a marked reduction of piston deposits and ring-sticking, but many addition agents caused corrosion of non-babbitt bearings. Extensive research resulted in the development of a non-corrosive lubricant which gave excellent results in a prolonged ring-sticking test. The same compounded oil showed a low rate of cylinder wear under both high- and low-temperature operating conditions; in comparison, the straight mineral oil permitted much greater wear, whilst some other compounded oils were not uniformly satisfactory at all temperatures.

Fundamental investigation shows that conventional oils tend to spread from hot to cold surfaces with a measurable force which varies with the temperature. An apparatus has been built for the study of "limiting adhesion temperature," and a correlation established between this characteristic of an oil and cylinder wear. It is believed that the piston-ring temperatures in many diesel and aircraft engines exceed the limiting adhesion temperatures of oils commonly used. The wear studies have shown that measurable differences exist between different lubricants in respect of ring-groove as well as cylinder wear. The former is important, since worn grooves allow oil-pumping and excessive blowby. Severe breaking-in tests resulted in scuffing of rings and pistons when a normal oil was used, whereas a compounded oil was quite satisfactory.

Anti-ring-sticking agents may be either oxidation catalysts or oxidation inhibitors. Both types may prevent ring-sticking, but the catalysts decrease the oxidation stability of the base, causing filter clogging and corrosion. A suitably compounded oil promotes filter cleanliness. The life of a compounded lubricant is dependent on the rate at which the compounding material is consumed. When regular oil changes were eliminated, auxiliary filters of the clay or Fuller's-earth type showed no advantage. The frequency of oil-changing should be determined in practice by operating conditions. Over an extended period of service oil consumption is primarily dependent on: (1) degree of freedom of the rings; (2) amount of carbon found in the oil-ring slots; (3) piston-ring wear; and (4) the surface condition of rings and liners. For this reason a compounded lubricant may prove more economical than a straight mineral oil costing less per gallon.

Diesel lubricants containing anti-ring-sticking agents may be employed to free piston-rings and remove piston-deposits that have been caused by previous use of unsuitable oils. This "purging" effect results in the gummy and carbonaceous materials which are removed from the pistons becoming suspended in the oil. Attention must therefore be given to the filters during initial operation with a "purging" oil.

The author claims that a compounded diesel lubricant is now available having all of the following properties: (1) anti-ring-sticking value; (2) non-corrosivity; (3) low wear at both high and low temperatures; (4) good breaking-in properties, and (5) high stability in both storage and service. K. T. A.

See also Abstract Nos. 348, 351.

Detonation and Engines.

362. Sound- and Pressure-Waves in Detonation. C. E. Grinstead. *J. Aeronaut. Sci.*, 1939, 6 (10), 412-417.—This paper presents studies of the relationship between the frequency of pressure-waves in the combustion chamber and the audible sound in air produced by detonation. Three engines were investigated: a two-stroke o.h.v. diesel, a modified "L" head Delco gasoline engine, and a variable-compression knock-testing gasoline engine. Sound and pressure waves were picked up with a microphone and carbon-stack indicator, respectively, and recorded by simultaneously photographing the screens of two cathode-ray oscillographs. The microphone was located in several different positions to find the point of maximum sound intensity.

Some of the records are reproduced, and it is shown that there is a one-to-one correspondence between the frequency of the shock-waves in the combustion chamber and the frequency of the audible sound in air. On the Delco engine the frequency of the shock-waves was increased from 3100 to 3415 c.p.s. by the addition of oxygen; it was found that there was a corresponding change in the sound frequency. Other interesting features of the records are discussed. K. A.

363.* Pumplless Injection. Anon. *Auto. Engr*, 1939, 29, 331.—A short description of a new type of diesel engine developed in Germany by the late Dr. Ing. Prosper L'Orange which has been successfully demonstrated as a 2000-c.c. two-stroke and also as small two-stroke and four-stroke engines having swept volumes from 100 to 300 c.c. The system employs the pressure differential between the main combustion space in the cylinder and an antechamber.

On the suction stroke fuel is admitted to a delivery channel by a mechanically operated valve. This channel is situated in an inserted block forming the throat between the combustion space and the antechamber. Small exit channels lead from the delivery channel to each side of the restricted throat. A thermally isolated heat-storage member is provided in the antechamber and a heater plug is fitted for starting purposes. The pressure difference between the ends of the two exit channels carries the fuel from the delivery channel to the antechamber and the main combustion space at different stages of the working cycle. During the compression stroke fuel is forced into the antechamber, but when combustion commences in the antechamber, with a consequent rise in pressure, the fuel flow is reversed, and the remaining fuel is delivered into the main combustion space. Tests were made on a 300-c.c. four-stroke engine developing 4 B.H.P. at 2400 r.p.m. equivalent to a B.M.E.P. of approximately 73 lb. per sq. in. Operating on a compression ratio of 19-1, the fuel consumption at the above power output was 0.5-0.55 lb. per B.H.P./hr. It is stated that a small 100-c.c. air-cooled two-stroke engine has been operated successfully up to 5000 r.p.m. C. H. S.

364.* Italian Multi-fuel Engine. Anon. *Auto. Engr*, 1939, 29, 358-360.—A description of a new type of engine now being fitted to the Fiat tractor which has been patented by Professor Boghetto.

The engine is of the injection type, with a comparatively low compression ratio of 7.2-1. It is fitted with high-tension ignition, and its main working characteristic is that it takes a full air charge at all loads up to one-sixth of the maximum load, and therefore its consumption at low loads follows the same consumption law as that of the diesel engine.

The basic principle of this engine is the stratification of the fuel mixture in a chamber which may be considered as separate from the power cylinder. This combustion chamber is so placed that it is not subject to the influence of the passage of suction air or exhaust gases, and it remains full of burnt gases at the end of the suction stroke. Fuel is injected into the combustion chamber in the direction of the mouth of the chamber, where it meets air coming from the cylinder and produces a homogeneous explosive mixture without any artificially created turbulence. The sparking-plug situated practically at the upper limit of the carburated air zone provides ignition as in an ordinary carburation engine. Data are given which show the performance of the engine on gas oil and 93° alcohol. On gas oil the consumption figures are comparable with those of a good diesel engine. The lowest specific consumption is obtained

at $\frac{1}{2}$ load, and at $\frac{1}{3}$ and $\frac{1}{4}$ load the consumptions are respectively 19% and 42% higher than that at full load.

C. H. S.

365.* French Multi-fuel Engine. Anon. *Auto. Engr.*, 1939, 29, 369-370.—A description of the Brandt-Bagnulo engine and Brandt-Bagnulo cylinder-head which can be supplied for converting existing Ford, Fordson, Citroen, Latil, and other French engines to this system. The efficiency of the diesel engine is claimed, combined with the light weight, flexibility, simplicity, and ease of maintenance of a petrol engine. It has the outstanding advantage of functioning either on gas or on light and heavy liquid fuels.

In the head-casting there is provided for each cylinder a special antechamber in which carburation, gasification, and the initial combustion of the fuel take place. The antechamber is in open communication with the working cylinder by means of a tangential passage. Fuel and air from a jet-type mixing device are admitted to the antechamber by a supplementary valve actuated through a relay lever from the normal inlet-valve rocker. The mixing device has two constant-level float-chambers to permit the use of two fuels, either separately or simultaneously. On the suction stroke fuel enters the antechamber simultaneously with the admission of pure air to the working cylinder. The fuel is drawn from the constant-level chamber and vaporized by a current of air from a diffuser as it leaves the jet. It then traverses heated conduits and enters the antechamber by the supplementary valve, and is directed towards a heated tongue in the vicinity of the sparking-plug, and is thus gasified before being mixed with air from the working cylinder. In this manner the spark always occurs in a highly explosive mixture, irrespective of the quantity of fuel introduced. For the Brandt-Bagnulo engine with a compression of 9-1 it is claimed a petrol consumption of approximately 0.44 lb. per H.P./hr. can be maintained at all normal working loads.

C. H. S.

366.* Combustion. H. Rabezana, S. Kalmar and A. Candelise. *Auto. Engr.*, 1939, 29 (389), 347-353 (390), 377-381.—Combustion phenomena have been studied in L-head engines; pressure development and flame movement were simultaneously recorded by photographing oscillograph diagrams. Pressure-time records were obtained by means of a carbon-stack element, and flame records by the ionization-gap method. The results have been analysed mathematically by a step-by-step method. Briefly, this consists of burning a volume increment at constant volume and expanding the same into the already-burned and still-unburned portion of the mixture, until pressure equilibrium is reached. An example of this analytical method is given.

Records obtained from various combustion chambers and under various operating conditions are analysed to show the effect of different variables. It is shown that the duration of the entire combustion cycle, depending mainly on the reaction velocity, controls the peak pressure and also detonation. From the experimental results it appears that the initial stage of combustion is the most sensitive to the effects of the various factors governing the reaction velocity; consequently it is the logical point of attack for efforts to produce a more stable and faster-burning mixture—i.e., less stratification and more turbulence in the vicinity of the spark plug at the instant of ignition.

K. A.

367.* Trends in Design of 1940 Cars. T. A. Bissell. *J. Soc. Auto. Engrs.*, 1939, 45 (5), 457-470.—A survey of engineering improvements in American cars for 1940. Progress is most marked in the transmission field, the most interesting development being the Oldsmobile four-speed automatic gear-box embodying a fluid flywheel. Auxiliary over-drive gears are available on a number of cars.

Engine compression ratios are still rising, 6.5:1 being quite common. Many carburation systems have been modified to give increased power; larger fuel pumps and insulated fuel lines have made their appearance. The general tendency is to increase engine powers by taking full advantage of the most recent improvements in fuels.

K. A.

Economics and Statistics.

368.* **Transport of Roumanian Oil by the Danube.** Anon. *Rev. Pétrolifère*, 29.9.39 (856), 1201; 28.10.39 (860), 1269.—95% of the petroleum products which are exported from Roumania by the Danube route are loaded at Giurgiu from storage reservoirs into tankers. Giurgiu is 900 miles from Vienna and 1170 miles from Regensburg in Bavaria. At the outbreak of war the Danube tanker fleet comprised 298 vessels, with a total capacity of 201,315 tonnes. This capacity has recently been increased by 20,000 tonnes. Germany owned 45% of this total capacity. Prior to July 1939 the greater part of German imports from Roumania were transported by sea from Constanza. The following statistics are given :

Exports of petroleum products from Roumania.

	1938. tonnes.	1939 (Jan.—June). tonnes.
<i>By sea from Constanza</i>		
To Germany and Austria	558,857	270,963
To Czechoslovakia	nil	121,083
	<u>558,857</u>	<u>392,046</u>
<i>By Danube Route</i>		
To Germany and Austria	134,407	157,750
To Czechoslovakia	294,898	131,029
	<u>429,305</u>	<u>288,779</u>

The round trip from Giurgiu to Germany and back, possible during nine months of the year only, takes five weeks. But this is dependent on appropriate storage or railway transport being available at the ports of loading and discharging. If these conditions were fulfilled, and the German tanker fleet was increased to 160,000 tonnes capacity, it might be possible for Germany to import 1,300,000 tonnes annually by the Danube route.

Some years ago, Russian oil, unloaded at Sulina on the Black Sea, was also transhipped to Danube tankers and imported by this route to Central Europe.

S. J. A.

369.* **Pre-War Expansion in German Motor Traffic.** Anon. *Petroleum Press Service*, 8.12.39. 6. 47, 571.—During the twelve months ended 1st July, 1939, motor vehicles registered in Germany exceeded those of the previous twelve months by about 16%. The following figures are given :

	1938.	1939.
Private cars	1,305,608	1,486,451
Goods vehicles	400,288	442,036
Omnibuses	20,792	23,302
Motor-cycles	1,582,872	1,860,722
Tractors	54,943	82,077
	<u>3,364,503</u>	<u>3,894,588</u>

These figures include vehicles in use in Austria. If the vehicles registered in Czechoslovakia are added, it is estimated that Germany can draw upon over 4 million vehicles.

S. J. A.

370.* **Production and Circulation of Automobiles Throughout the World.** Anon. *Monit. Petr. roum.*, 1939, 40, 1471-1476.—This article is a mass of statistics for the year 1938. Every country is tabulated, with the number of automobiles registered,

manufactured, and exported; the population and persons per vehicle; the length of the road systems, and the consumption of motor spirits. The greater part of the figures appear to have been extracted from the annual publication of the American Automobile Manufacturers Association, *Automobile Facts and Figures*.

In 1938 in the U.S.A. there were 29,490,728 vehicles, or 1 to every 4 persons. Great Britain had 2,422,675 vehicles, with 20 people per vehicle, whilst the figures for other countries were: France 2,233,500 automobiles, 19 people per vehicle; Germany 1,753,600 and 45; Italy 469,400 and 92; Roumania 35,500 and 563; Russia 672,952 and 251; Japan 180,800 and 395; Australia 799,750 and 9; South Africa 349,631 and 28; and China 45,196 and 9,957. The greatest number of people per vehicle is found in Liberia.

A few examples of the length of roads in kilometres are: U.S.A. 4,937,477; U.S.S.R. 2,706,513; Japan 1,070,619; France 633,561; Germany 416,364; and Great Britain 308,130.

In 1938, 4,001,566 automobiles were produced, 62.2% by U.S.A., 11.2% by Great Britain, 8.8% by Germany, 5.3% each by France and Italy, and 4.1% by U.S.S.R.

89,909,000 tonnes of motor spirit and 8,500,000 tonnes of lubricating oil were consumed by automobiles throughout the world.

T. C. G. T.

371.* Statistics on the Personnel of the (Roumanian) Petroleum Industry. Anon. *Monit. Petr. roum.*, 1939, 40, 1297-1301.—The personnel of the Rumanian petroleum industry in 1938 has been analysed and tabulated according to occupation, employers and location.

31,600 people are employed in the industry, 49.7% in the field and 25% in refineries. Of the major companies, Astra Romana head the table, employing 6352 people, 46.9% in the field and 28% in refineries. For Steaua Romano, the figures are respectively 4048, 63.4% and 24.5%; Concordia 3426, 49.8% and 26.3%. Unirea 3323, 40.2% and 17%; Romano-Americana 2890, 36.5% and 48.3%. It is also of interest to note that Petrol Block employed 399 people in 1938, 80.9% being in refineries.

The total of 15,700 employed in the field compares with 18,430 in 1937, whilst there has also been a drop from 8200 in 1937 to 7900 in 1938 in the refinery employment.

T. C. G. T.

372.* Production of Crude Oil in Venezuela in March 1939. Anon. *Rev. del Min. Fomento (Venezuela)*, June 1939, II (13), 616-623.—Total production in March 1939 from the oil-fields of Venezuela amounted to 2,323,149.926 metric tons, showing an increase of 121,407.435 metric tons over the preceding February. This difference is due to the smaller number of days in that month. The actual daily outputs for the two months were 74,940 metric tons in March and 78,632 metric tons in February, showing a difference of 3693 metric tons in favour of the latter month.

Compared with March 1938, this year's output shows an increase of 89,416.765 metric tons, this being due to the outputs of three new fields, at Bachaquero, Temblador, and Oficina.

Total export in March by the oil companies was 2,262,111.148 metric tons.

During this month seven of the leading companies refined 127,395.894 metric tons.

New Wells. Twenty-two were started up: twenty-eight came into production, thirty-three were in process of being drilled.

In addition to the above, a number of trial borings were made in the Eastern region of Venezuela. These reached depths up to 3000 metres approximately. Out of ten wells, one reached the production stage at 1841 metres and one was abandoned. Drilling continues in the remainder.

In the Central region one well is being sunk, and in the Western region one well has reached a depth of 2641 metres, and samples are being obtained.

H. I. L.

373. Particulars of Oil Concessions in the State of Monagas, Venezuela. Anon. *Rev. del Min. de Fomento (Venezuela)*, July 1939, II (14), 129-130, August 1939, II (15), 381-382, September 1939, II (16), 531-532.—Tables are given showing all essential particulars of the oil concessions granted in the State of Monagas since the commencement of production, including the names of the concessionaire, the area of the fields, and the duty paid thereon. Earliest demands for concessions were made in 1929.

Growth of Production.—A graph is shown giving the growth of production in Venezuela from nothing in 1917 up to 2,680,000 metric tons in July 1939. H. I. L.

374. Argentine Petroleum Industry (Action taken by the Republic). M. L. Villa. *Bol. Inform. Petroleras*, October 1939, XVI (182), 5-66.—A fully detailed historical and statistical review of the development of the Argentine petroleum industry as operated by the Government since its inception, showing the great progress made by the setting up of the Y.P.F., whose aim is to develop the resources of the country for the benefit of the community as a whole, as opposed to the work of private concerns. Figures given are eloquent of the success of the scheme, which was initiated in 1910. The article is well illustrated and interesting graphs are given. H. I. L.

375. Petroleum in Venezuela. Anon. *Bol. Inform. Petroleras*, October 1939, XVI (182), 67-87.—An account of the present position, with statistics, of the petroleum industry in Venezuela. In 1938 production reached the considerable figure of 27 million metric tons. In spite of this, there is urgent need for financial reform. Most of the oil is exported, and production is in the hands of big American and European companies. The Government appear to be desirous of emulating the action of the Argentine Republic, but whereas the latter country is capable of consuming their entire output, conditions are reversed in Venezuela. H. I. L.

376. World Petroleum Legislation. Anon. *Bol. Inform. Petroleras*, October 1939, XVI (182), 88-97.—The notes on World Legislation on Petroleum, which have appeared regularly since No. 174 of this journal, are of particular interest in the present number, as they deal exclusively with Mexico. The course of the graph (p. 89) showing the annual production from 1901 to 1938 is of peculiar interest. H. I. L.

BOOK REVIEWS

Recent Marine Sediments. A symposium edited by P. D. Trask. Pp. 736. 137 figures. Published by the American Association of Petroleum Geologists, Tulsa, Oklahoma, U.S.A.; London, Thomas Murby and Co. Price \$5.

Sediments are of pre-eminent interest to oil geologists, and it is therefore appropriate that "Recent Marine Sediments" should be published by the American Association of Petroleum Geologists. However, its appeal is not limited to oil geologists.

Some years ago the Committee on Sedimentation, under the direction of W. H. Twenhofel, sponsored a Treatise on Sedimentation which dealt comprehensively with both recent and ancient sediments. The new symposium is intended as a supplement to the Treatise. Its various articles have been contributed by American, European, and Japanese authors, and cover a wide range of subjects. These are grouped under the following headings: transportation; the relationship of oceanography to sedimentation; deposits associated with the strand line; near-shore sediments—hemipelagic deposits; pelagic deposits; special features of sediments; methods of study. In addition to descriptions of the distribution of various types of sediments, information is given on the physical processes in the ocean, rates of sedimentation, the occurrence and activity of bacteria in marine sediments, the organic content of recent marine sediments, the properties of clays, faecal pellets in relation to marine deposits, mechanical, mineralogical and X-ray analyses, bottom-sampling apparatus, and the presentation of sedimentary data.

Each paper is the work of a specialist, who has summarized the progress in his particular field, and since some subjects are discussed by more than one author, different explanations are offered for certain phenomena.

Palæogeographical considerations are becoming more and more important in the search for oil, and consequently all data derived from a study of recent marine sediments which facilitate the interpretation of ancient sediments are especially welcome. Furthermore, the studies have a bearing on the perennial problem of the origin of oil. The physical geologist, too, will find much of interest in this volume, and some papers may well be read by the petrologist and geochemist.

Extensive and up-to-date as is the information presented, much of the value of this symposium lies in its being a key to a wide range of literature on the different topics, for each paper is rounded off with a list of selected references. The accessibility of the contents of the symposium is made very easy by a subject index sixty-two pages in length.

G. D. HOBSON.

A Practical Manual of Chemical Engineering. By Harold Tongue, M.I.Mech.E., Mem.A.S.M.E., A.M.I.Chem.E., with a foreword by Sir Gilbert Morgan, O.B.E., D.Sc., Sc.D., LL.D., F.R.S. Pp. xv + 560. Chapman & Hall, Ltd., London. Price 36s.

The object of a text-book dealing with any branch of engineering is generally to present the fundamental scientific principles and to indicate how these principles are applied and can be applied in practice. This desirable combination is not always achieved. The scientific principles may be well set out without it being clear to the reader how they can be utilized to advantage in dealing with practical problems. On the other hand, books which emphasize the practical aspect often take the form of collections of tables and data or descriptions of plant in ordinary use. Although valuable in their sphere, such books fail to bring out the principles by which practice is, or should be, guided. Judged by these criteria, Mr. Tongue's book has attained a high standard. In it the principles underlying chemical engineering operations are treated on similar lines to those which have been established by the pioneer

American text-books in this field, though naturally not in the same detail; the practical aspects of these operations are emphasized to a greater degree without losing sight of the basic principles.

Materials of construction, both metallic and non-metallic, which are such an important subject in chemical engineering practice, are dealt with at some length. This section, which also contains much useful information on methods of fabrication, occupies about a third of the book. The other chapters deal with the design and construction of pressure vessels, pipes and pipework, heat insulation, steam plant for chemical works, fluid flow, heat transfer, evaporation, drying, adsorption, distillation, filtration, crushing and grinding, and pumping. The field is a wide one, and Mr. Tongue has covered it well. His book will be of great value to those whose work is concerned with chemical engineering. A. J. V. UNDERWOOD.

The Cracking Art in 1938. U.O.P. Booklet 239, edited by Gustav Egloff with the co-operation of Mmes. Crandal, Doty, and Jordan and Messrs. Dryer, Johnstone and Hulla, indexed by Mme. Laetz. Pp. 458. Universal Oil Products Co., Chicago.

The petroleum industry is very familiar with the information and up-to-date brochures sponsored by the Universal Oil Products Co. and edited by our distinguished American member, Gustav Egloff.

The present volume is a stupendous achievement, and it illustrates in an astonishing fashion the vast volume of research poured out in the short span of one year on the outstanding problem of the up-grading of mineral oil.

For that is the real objective before the industry. Natural oil from the well is woefully unfitted for our modern purposes. At every stage its fractions, once admittedly suitable and sufficient for the demands of civilized men, are found to be crude, ill-balanced, immature, incomplete, and in point of fact needing drastic revision.

And so we can contemplate the time when our familiar crude oil will be split up into chemical bricks from which we can fashion the special structure we need, be it gasoline, fuel, or lubricant. Although this time has not quite arrived, Egloff's summary of progress in one short year indicates that it is on the way.

The volume before us details most of the outstanding achievements of 1938: the development of catalytic cracking; the development of the so-called "midget" polymerization unit; the development of *iso*-octane units involving a double catalysis; the outstanding development of alkylation units which produce *iso*-octane and its homologues from the straight combination of *iso*-paraffins and olefines; the development of new methods of refining and the greater use of anti-oxidants.

Egloff stresses the immediate importance of catalytic cracking combined with the decomposition of the inherent heavy residue. He emphasizes the vast importance of research on the chemistry and physics and thermodynamics of the gaseous hydrocarbons, and indicates how, from these sources, new chemical "bricks" may be produced for synthetic purposes.

To give even a brief and inadequate summary of this most valuable publication would be a task of great difficulty, but the chapter-headings will perhaps suffice:—Statistics—catalytic research on cracking—pyrolytic research on cracking—electrical methods of research—survey of commercial cracking operation—equipment for cracking plant, tubes, furnaces and heat exchangers—corrosion—treatment of cracked products—inhibitors—by-products, alcohols, glycols, esters, halides, sulphides, etc., from cracked gases and a general discussion on the production of high-octane aviation fuel by means of polymerization, alkylation, dehydrogenation, ring closure, and hydrogenation.

The work finishes with a thoroughly comprehensive abstract of the patent literature, produced, as all the petroleum world knows, with the meticulous care and accuracy that characterize the library service at Riverside. Your reviewer, with many happy memories of this great research station, of Dr. Egloff and his associate, Prof. Ipatieff, and the staffs of the laboratory and the library, wishes to take this opportunity of thanking them for a great contribution to our knowledge.

A. E. DUNSTAN.

Modern Road Emulsions. By F. H. Garner (Editor), L. G. Gabriel and H. J. Prentice. 2nd Edition, 1939. Pp. xv + 245. The Road Emulsion and Cold Bituminous Roads Association Ltd., London. Price 10s.

This book started as a series of lectures to road engineers. These lectures were expanded and published as a book in 1933: there now appears a second edition, in which previous omissions have been made good and additions and modifications necessitated by increased knowledge and experience have been made.

Those closely conversant with the subject will find the book rather elementary, but those who seek to know the essentials of the subject and to receive practical advice will find it here, pleasantly set out. The style of writing is simple, and the illustrations are adequate and interesting, although some have been seen some time ago.

Roughly, the first half of the book is concerned with the road emulsions and their uses, whilst the second portion includes tests and specifications, British and foreign. There are two appendices on the Determination of the Size-Frequency Curve of Emulsion Particles, and Average Diameter of Particles; a Glossary of Terms, an excellent Bibliography, and a collection of twenty-six Tables of great practical interest.

The "get-up" of the volume makes it more of a "book" than was the first edition, and in all aspects it is a pleasant technical companion on one's shelf.

P. E. SPIELMANN.

INSTITUTE NOTES.

FEBRUARY 1940.

CANDIDATES FOR ADMISSION.

The following have applied for admission to the Institute or transfer to another grade of membership, and in accordance with the By-laws the proposals will not be considered until the lapse of at least one month subsequent to the issue of this *Journal*, during which any Fellow, Member or Associate Member may communicate by letter to the Secretary, for the confidential information of the Council, any particulars he may possess respecting the qualifications or suitability of any candidate.

The object of this information is to assist the Council in grading candidates according to the class of membership.

The names of the candidate's proposer and seconder are given in parentheses.

ALLCARD, Harry Ganly, Director (*H. G. Allcard & Co., Ltd.*), "Ravenswood," Wythanshawe Road, Northenden, Manchester. (*J. E. Haslam; A. McCulloch.*)

ANDERSON, William Archibald, Information Officer (*N.S. Wales Government Railways*), 19, York Street, Sydney, N.S.W. (*O. Colverd.*)



CARTER, Edward Howard, Transport Manager (*G. & T. Earle, Ltd.*), "Beaumaris," 44, Carr Lane, Willerby, E. Yorks. (*J. E. Haslam; H. S. Kernan.*)

FORSTER, Denis Williams, Chemist (*J. White & Sons, Ltd., Widnes*), "Woodlea," Brookside Avenue, Sankey, Nr. Warrington, Lancs. (*A. McCulloch; J. E. Haslam.*)

MOND, Albert Levy, Chemical Engineer, 14/18, High Holborn, London, E.C.1. (*V. Henny; E. N. Hague.*)

PARTRIDGE, William Arthur, Chemist (*Anglo-Iranian Oil Co.*), 10, Norman Court, Upper Sunbury Road, Hampton, Middx. (*A. E. Dunstan; F. B. Thole.*)

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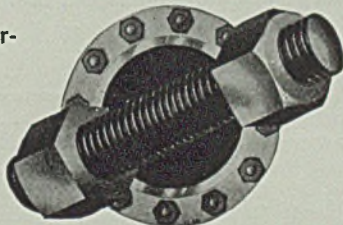
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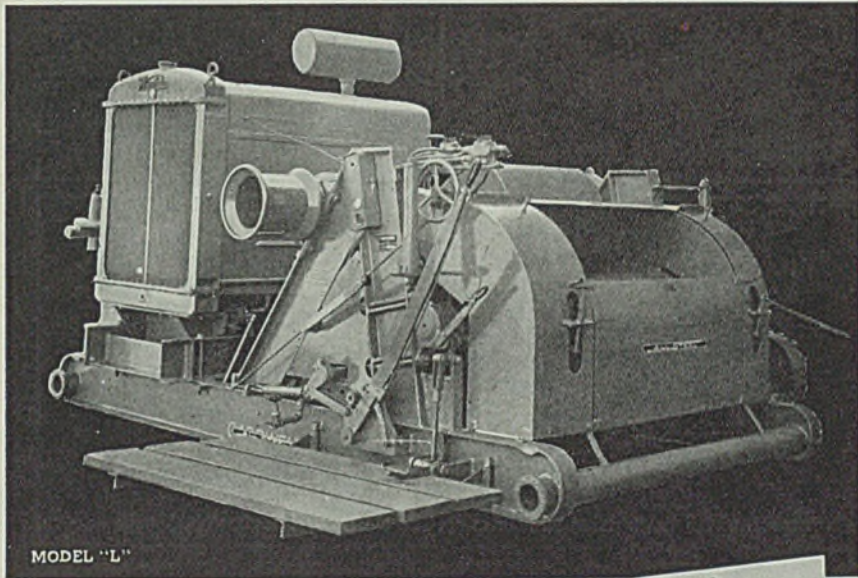
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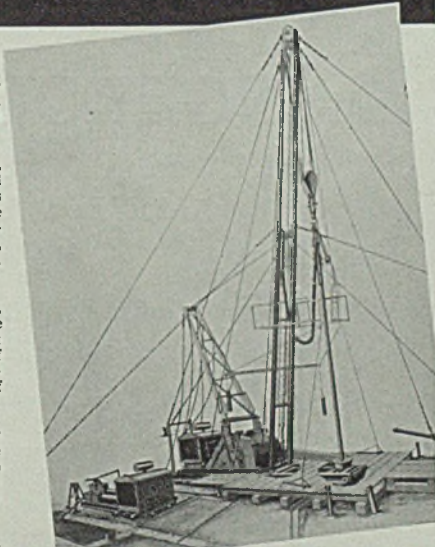
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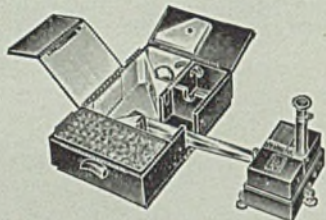
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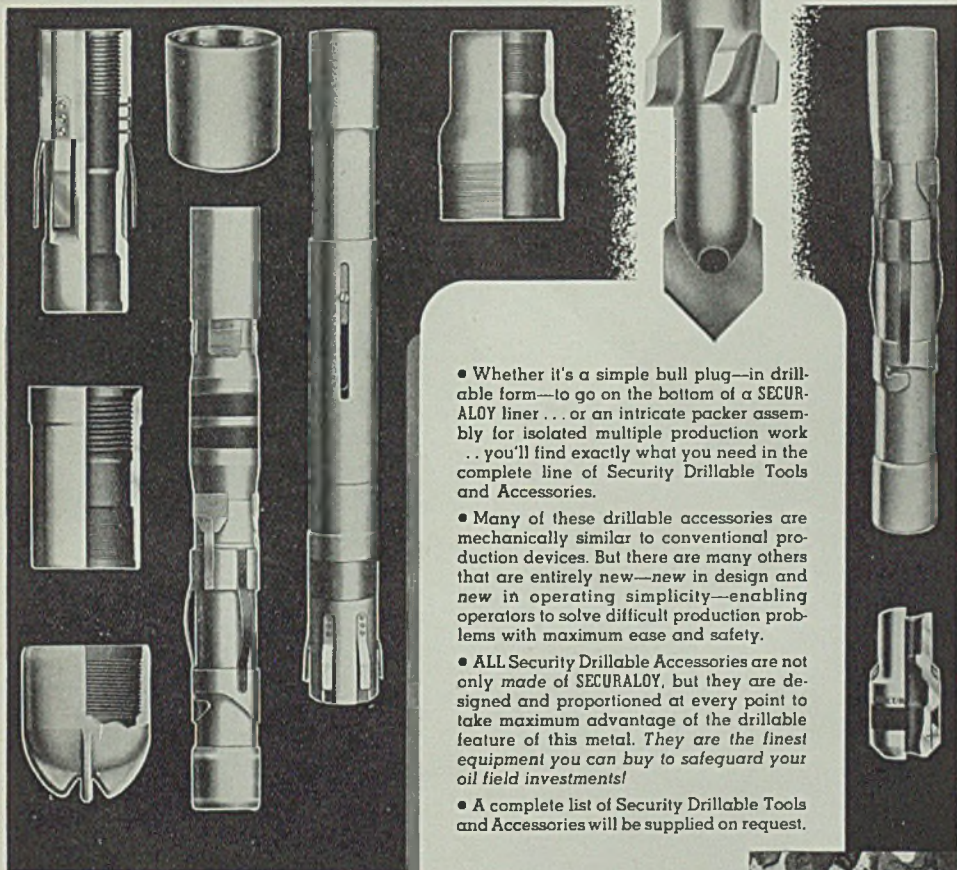
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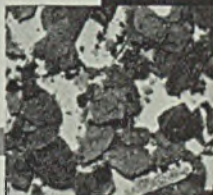
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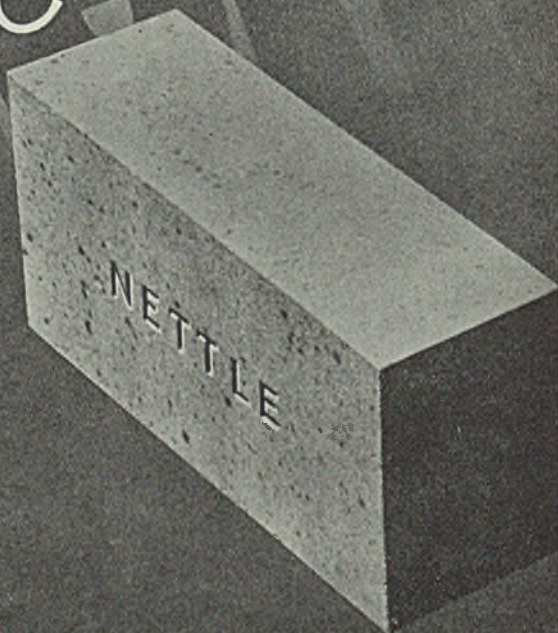
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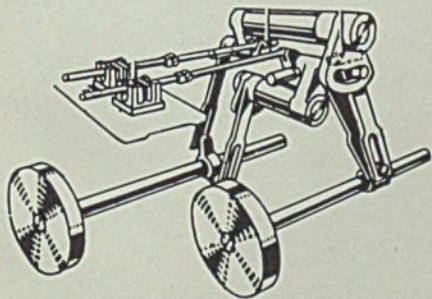
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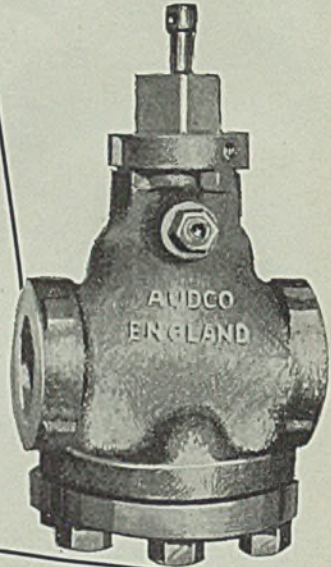
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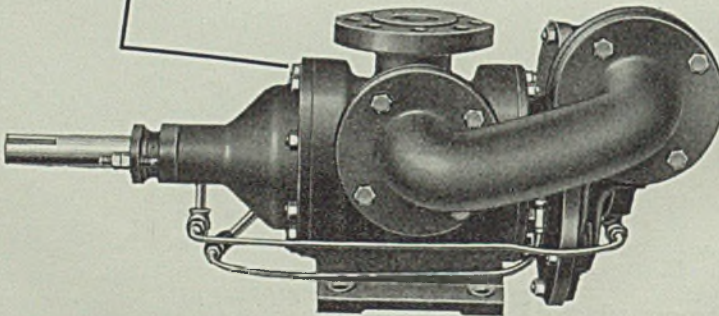
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