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CONTROL OF DRILLING MUD IN THE FIELD.*

By H. C. H. Darley, B.Sc., A.M.Inst.Pet.

Introduction.

The laws governing the chemical and physical properties of mud are not yet fully known, and much research work continues to be done on the subject. Most of us on the field have little time for research and must apply the results of others to the practical problems encountered in handling mud. This paper therefore formulates no new theory, but aims at presenting a means of testing muds which will give rapid and comparative results, and at establishing from these some system of treatment and control.

Flow Characteristics of Mud.

The essential flow characteristics of mud were clearly demonstrated by P. H. Jones and E. C. Babson [1] using a MacMichael type viscometer. With this type of viscometer mud is rotated at various speeds in a cup. An open-ended cylinder is suspended in the mud on a torsion wire. The deflection of this cylinder gives a measure of the resistance to shear of the mud at any instant.

Very briefly their results were as follows :—

If a thixotropic mud is sheared at a given speed, it will gradually assume a deflection typical of that speed, irrespective of its previous state of shear. This value is attained when equilibrium is established between the gelling and disrupting forces. If a mud is sheared to equilibrium at a given speed and the rate of shear then reduced, the initial deflections at each successive lower rate of shear will give a straight line which will, if projected, cut the axis above zero. The slope of this line is called by these workers the " immobility," and the value at which it cuts the axis the " yield point " (see Figs. 1 and 2). Since they are determined before thixotropy can have any effect, immobility and yield point are typical of the rate at which the mud was originally sheared to equilibrium. To define the flow characteristics of a mud, yield point expressed in dynes/cm.^{2} and immobility in centipoises for several rates of shear are given.

The author made some similar but more approximate experiments on drilling muds taken from Apex Wells. A synchronous motor was used to stir the mud at a fixed rate by driving a paddle in a cup fitted with baffles. Two speeds were obtained through gears. The cup had a capillary tube fitted to the bottom. In the first series of experiments the length of time required to shear a mud to its equilibrium value at each speed was found by stopping the paddle every minute and timing the discharge rate through the capillary tube. The discharge rate was then plotted against length of time sheared. Over a number of muds the time required to reach approximate equilibrium was found to vary from 10 minutes to more than half an hour. Examples of each type are given in Fig. 3.

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* Paper read to a meeting of the Trinidad Branch on 1st May, 1940.

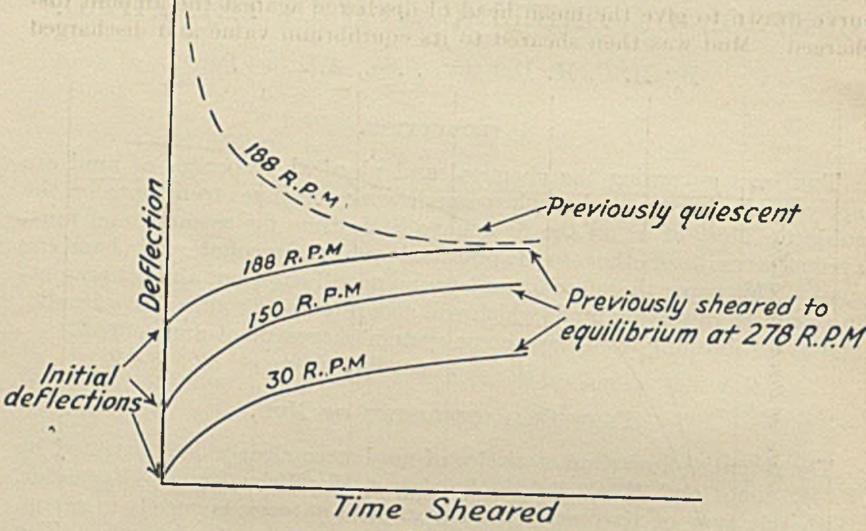
Deflection
188 R.P.M
Previously quiescent
188 R.P.M
150 R.P.M
Previously sheared to equilibrium at 278 R.P.M
30 R..P.M
Initial deflections
Time Sheared

FIG. 1.

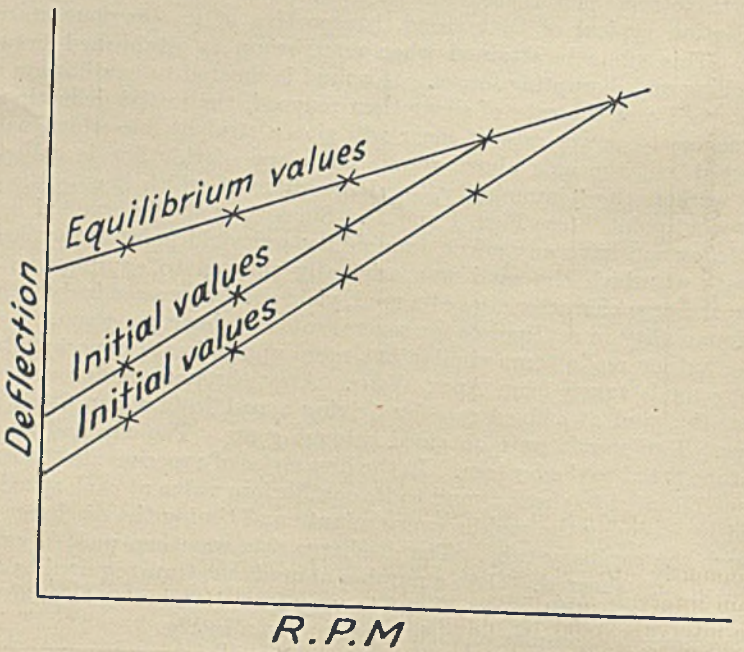
Equilibrium values
Initial values
Initial values
Deflection
R.P.M

FIG. 2.

In the second series of experiments the cup was first calibrated and a curve drawn to give the mean head of discharge against the amount discharged. Mud was then sheared to its equilibrium value and discharged

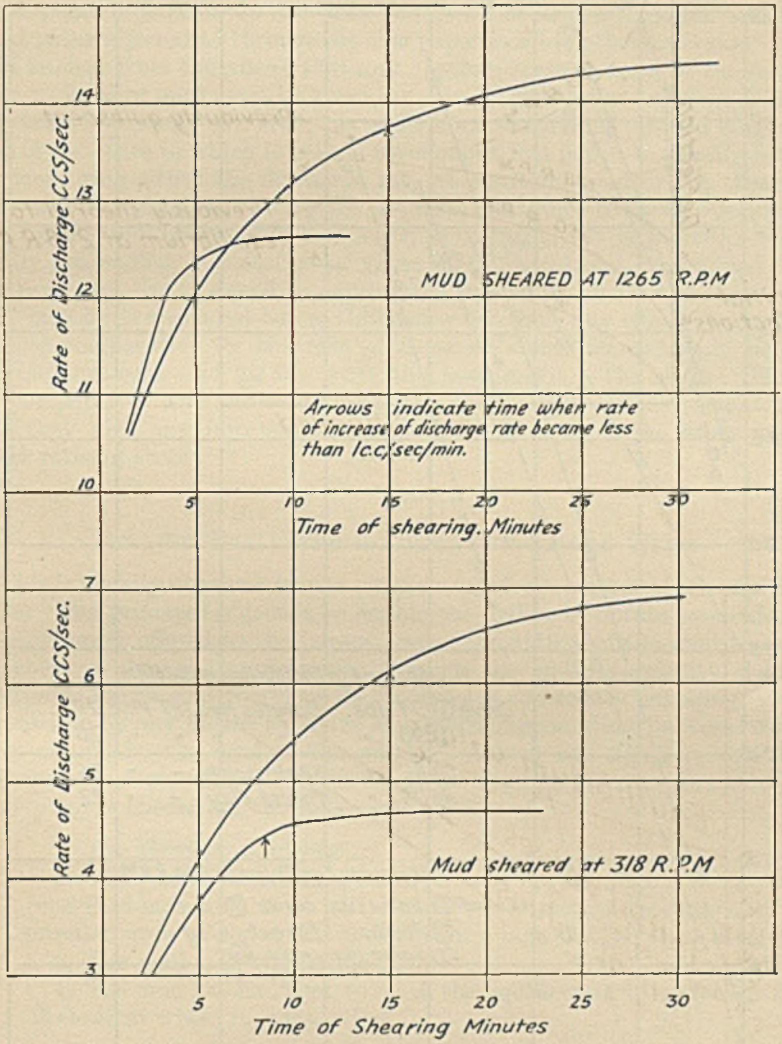

FIG. 3.

VARIATION OF VISCOSITY OF MUD WITH TIME OF SHEARING.

continuously into a graduated cylinder. The time at which the level passed certain intervals was noted, and thus the discharge rate between each of these intervals could be obtained and plotted against the mean head at which it was discharged. The whole cup seldom took more than half a minute to discharge, and thixotropy was consequently negligible. The method is rough, and it is necessary to take the mean of about six sets of readings to get reasonably accurate results.

Some typical results are shown in Fig. 4. The slope of a line is a repre-

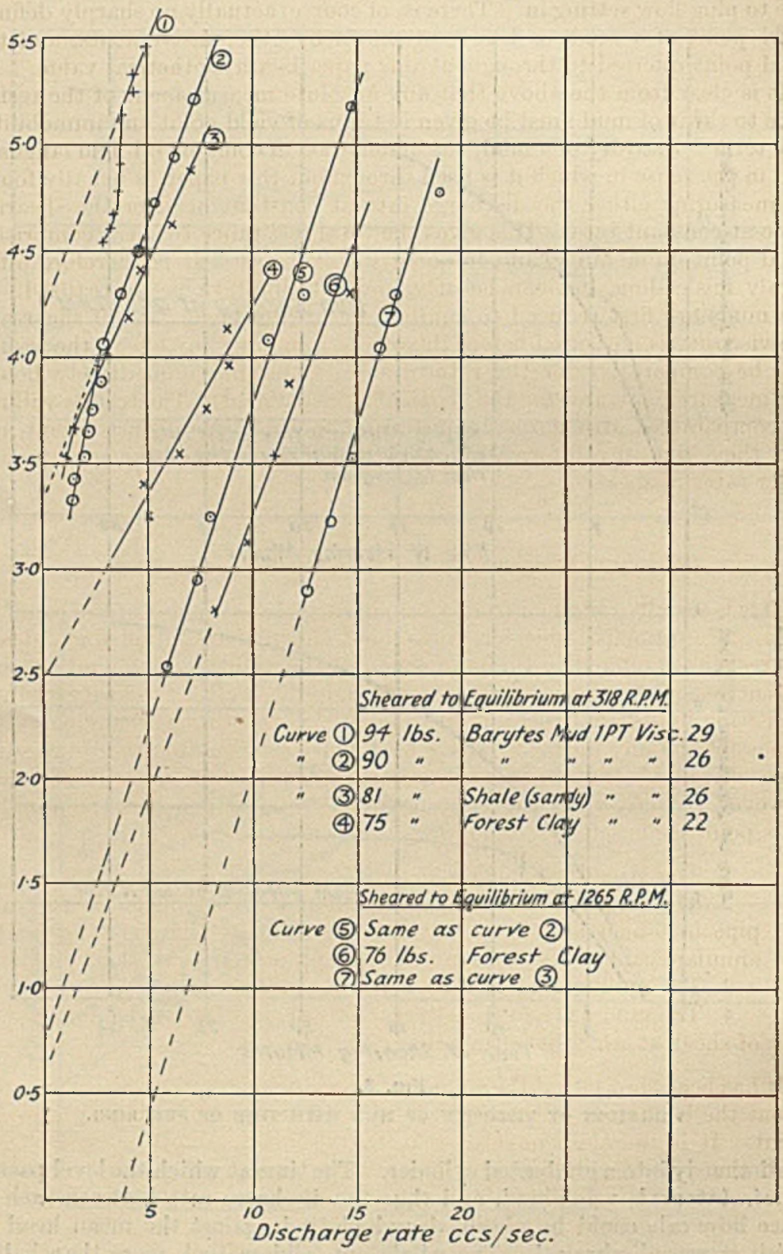

FIG. 4.

VARIATION OF RATE OF DISCHARGE OF MUD WITH HEAD.

sentation of the immobility. The point at which a line cuts the axis indicates the yield point for the previous rate of shear. It can be seen that at low rates of shear the curve leaves the straight line. This is probably due to plug flow setting in. There is, of course, actually no sharply defined yield point of a mud, as has been shown by other instruments, and the yield point referred to throughout this paper is a hypothetical value.

It is clear from the above that any absolute measurement of the resistance to shear of mud must be given in terms of yield point and immobility. The term " viscosity of a mud," as understood in common oil-field language and in the sense in which it is used throughout this paper, is usually found by measuring either the discharge rate at constant head or the shearing force at constant rate. This gives the total resistance to shear comprising yield point, immobility, and frequently thixotropy. It is therefore often highly misleading, but can be of value if its limitations are realized. If the muds are first reduced to equilibrium at any given rate of shear and the viscosity is measured before thixotropy can have any effect, the results will be comparative for the rate of shear obtaining immediately before the measurement and for the particular instrument. The results will not be comparative with those obtained from another type of instrument, nor will they give any information as to the behaviour of the muds under other rates of shear.

Routine Evaluation of Viscosity at the Well.

This is usually obtained from a simple rugged viscometer of the capillary type. The principal object is to enable the driller to obtain some idea of the resistance offered to the pumps and of the ability of the mud to carry up cuttings. As the mud travels through the well its velocity changes over wide limits, and therefore its viscosity is constantly changing. It is impossible by any means to arrive at a figure representing the total resistance offered by the mud to the pumps. A very fair figure is obtained, however, by measuring the viscosity of the mud as it leaves the annulus. This point is chosen for the following reasons :—

1. The viscosity is highest.
2. The resistance to shear in an annulus is very high. With 3-inch pipe in 6-inch hole Reid and Evans [2] estimate the resistance in the annular space at about six times that of a pipe of equivalent diameter.
3. The mud is in streamline flow.
4. The mud has had time to reach the equilibrium value for the rate of shear at which it is travelling.

It has been shown that the single-reading viscometer gives no information about the behaviour of the mud at any rate of shear but that of measurement. It is therefore obvious that the viscometer must be so designed that the velocities through the capillary correspond approximately to those obtaining in the annulus. (This must of necessity be approximate, since flow conditions in the capillary tube and the annulus have not yet been properly correlated.) Further, the sample must be taken and measured in a very short space of time if thixotropy is not to alter the results. The local I.P.T. viscometer * does not satisfactorily fulfil either of these requirements. It is somewhat slow in use and the velocity through the orifice appears to be on the high side. Because of the latter defect it is inclined to give comparatively low readings with high gel muds. The most obvious example of this is cement-cut muds. A reduction in head would remedy both these defects.

It is useful at the well to take a viscosity of the mud entering the well after degassing. The difference between the two viscosities is a rough measurement of the thixotropy.

ROUTINE VISCOSITY DETERMINATIONS IN THE LABORATORY.

The object of these is more to find out something of the properties of the mud with a view to controlling it, rather than the actual resistance to shear at the well. There are two opposing factors to be considered : time and accuracy of results. If time is no object, a viscometer like the MacMichael, giving both yield point and immobility, should unquestionably be used.

For quicker results a single-shot instrument may be used, but, as pointed out previously, the results are of limited value. The mud must first be reduced to equilibrium at some standard rate of shear. The most suitable rate of shear will be an approximation to that usually prevailing in the annulus, and viscosities through the capillary should be of the same order.

A suitable viscometer of this type is the capillary cup with the mud previously stirred by a motor-driven paddle. A gear-box will allow for two speeds, which can be selected by experiment to give rates of shear similar to " ingoing " and " outgoing " mud.

The largest source of error in this method is the correction for specific gravity of the mud. To obtain comparable results all readings must be corrected to a standard head. This cannot be done accurately unless immobility slopes are known. With similar types of mud such as may be obtained on one field, immobility slopes may differ little enough to allow a single slope to be assumed, and the correction then becomes :—

$$r = r_1 + (H - H_1) \tan. \theta$$

when r is discharge rate at head H
r_1 is discharge rate at head H_1
and θ is the immobility.

An approximation necessary on account of time is the equilibrium value, there being insufficient time to shear the mud until its viscosity is really constant. The mud must therefore be sheared until the rate of change of viscosity falls below a certain standard.

It can be seen from the above that with the limited time available for routine tests viscosity readings must remain an approximation with a limited application.

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* This is a viscometer of the Marsh type designed to meet Trinidad conditions as a result of discussion between interested members of the local I.P. Branch.

CLASSIFICATION OF MUDS.

The particles comprising a mud may be divided into two groups: suspensory, *i.e.*, those which would suspend themselves if isolated and made up into a separate suspension—and suspensoids, which are those that would not. This classification is somewhat indefinite, because the ability of particles to suspend themselves is affected by their concentration. Further, the division between suspension and deposition is by no means sharp, and time must always be taken into account. But while there is a group of particles the classification of which is uncertain, the great majority fall clearly into one or other of the groups.

If a suspensoid mud is diluted by two or three times its volume with water, then the particles which are immediately deposited are suspensoids. This analysis is rough, but simple and quick. The use of the term suspensoid is perhaps unfortunate in view of the fact that many workers use it to denote a hydrophobic sol.

The suspensory group is mainly composed of particles of colloidal dimensions. These can be classified as hydrophilic or gel-forming colloids and hydrophobic colloids. The distinction between the two is very marked. Hydrophilic colloids give a very much more rapid rise in viscosity with increasing concentration. They are also responsible for the thixotropy, including, of course, the formation of gels. It is therefore to be expected that they will have a predominating effect on the mud, and the proportion and properties of those present are the main factors in determining its behaviour.

The suspensoid group consists broadly of inert particles of clay, sand, and barytes of dimensions greater than colloidal. Their effect on the viscosity of a suspension is relatively small, but increases with their concentration until they eventually form a mechanical structure. The particle size of suspensoids largely determines their effect on the viscosity of a suspension. (See Reid and Evans [2]—" Drilling Mud.")

The behaviour of a mud can be controlled by its composition. The necessary viscosity for the primary functions is best supplied by gel-forming colloids, because of their thixotropic nature. On the other hand, too high a concentration is highly undesirable, because of the high viscosity involved. Suspensoids are only desirable in a mud to supply weight. Hence their specific gravity should be as high as possible. The greater their particle size the less their effect on the viscosity, but particle size is limited by two factors. In the first place, a balance must be struck between particle size and the concentration of gel-forming colloids necessary for suspension. In the second place, too large a particle size will cause too much abrasion.

Now, it has been seen that the viscosity of a mud is a somewhat difficult thing to determine, and even when determined it gives only limited information about the mud. If a simile may be used, a high viscosity is like the temperature of a sick man: it shows that he is ill, but does not say what is wrong with him. Since the properties of a mud must be determined by its composition, much fuller information about all aspects of its behaviour can be obtained from an analysis. The author has used a limited form of classification which has given good results with the similar types of mud such as are obtained from one field. In this system unloaded clay muds are classified according to the ratio of the viscosity to the percentage of solids by volume. This ratio is the essence of the classification, because, since the particle-size distribution of the suspensoids of the normal drilling muds is very much the same, it is an indication of the colloidal properties, the higher the viscosity for a given amount of solids, the greater the strength of the gel-forming colloids. It is also held that the action of the gel-forming colloids is the basic factor in determining the behaviour of the mud. A simple way to express results is to select some clay as standard and measure its viscosity at various concentrations. The viscosity of a test clay is then compared with these, and the concentration of the standard clay required to give the same viscosity as the test clay will be a comparison of their colloidal properties. Thus a 80–77 lb./cu. ft. clay means a mud weighing 80 lb./cu. ft. the viscosity of which is the same as a 77 lb./cu. ft. mud of the standard clay. Since these two figures give an expression for the concentration and nature of the suspension, quite a good opinion may be formed of its behaviour.

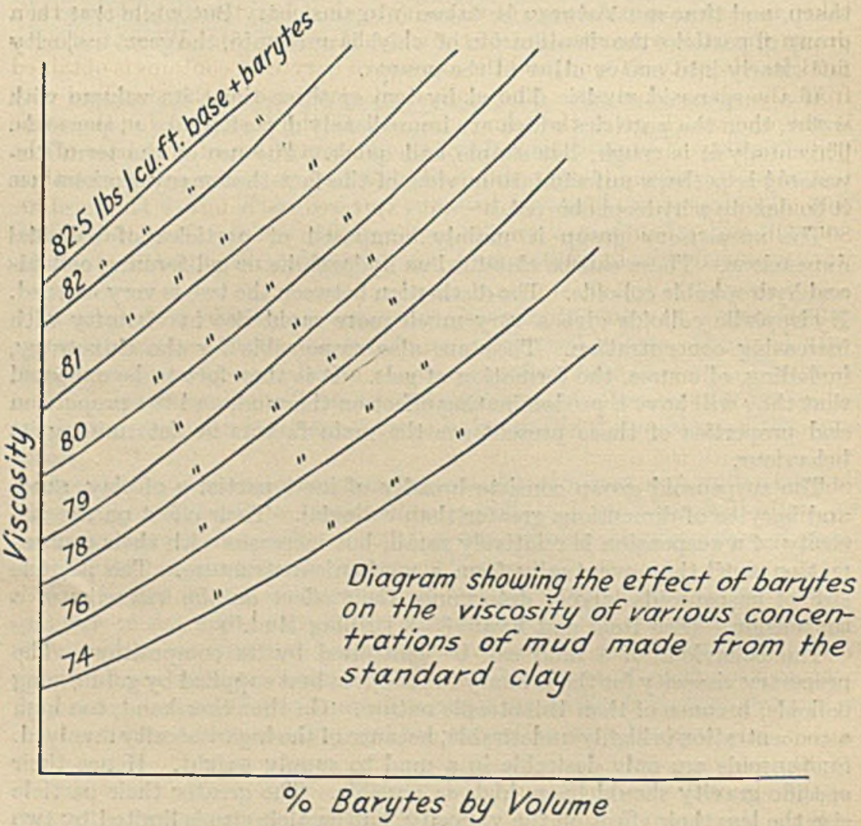

Diagram showing the effect of barytes on the viscosity of various concentrations of mud made from the standard clay

FIG. 5.

If the mud contains barytes it must be analysed and considered as two components, the clay mud as the base and the barytes. The mud is first evaporated to dryness and the specific gravity of the solids determined. The total percentage of solids by volume in the original mud is then calculated, and from it and the weight per cu. ft. the clay base can be easily calculated or obtained from a chart. The concentration of the clay base being thus determined, it remains to obtain a measure of its colloidal properties. In a previous experiment various concentrations of the same standard clay were loaded with various amounts of barytes and the viscosity taken in each case. A curve of viscosity against barytes content was then drawn for each concentration of the clay base (Fig. 5). When testing a mud, its viscosity is found, and the amount of barytes it contains is obtained from the above analysis. The equivalent clay base of the standard clay is the one which gives the same viscosity as the test mud for the same percentage of barytes. The ratio of the concentration of clay base of the test mud to the equivalent standard clay base is a comparison of their colloidal properties. The results are expressed as before. Thus a 100–80–77 lb./cu. ft. mud means a mud weighing 100 lb./cu. ft. If all the barytes were removed it would weigh 80 lb./cu. ft. and would have the same viscosity as a 77 lb./cu. ft. standard clay mud.

The author has used this system on daily samples from drilling wells with satisfactory results. The main difficulty is that, to yield comparative results, the muds should all be in the same electro-chemical condition, whereas, of course, the muds came in from the wells in varying stages of chemical treatment. The only way to overcome this difficulty is to treat the mud with the usual reagents and use the lowest viscosity that can be obtained. In preparing the curves, the standard clay was, of course, similarly treated. While this probably does not always give strictly comparative figures for the actual composition of the mud, yet as a practical means of predicting the behaviour of muds it works out very well.

For the sake of convenience in drawing graphs and compiling records the author uses the ratio of the percentage solids in the equivalent standard clay base to that in the test clay base as an indication of the colloidal properties. This ratio has been called the "colloidal index," and the higher it is the higher is the proportion of gel-forming colloids. It is not a very satisfactory term, because, if a mud is diluted and tested at various stages of dilution, its colloidal index changes. This is primarily due to the shape of the viscosity–concentration curves.

Since thixotropy also depends mainly on the amount of the gel-forming colloids present, it is reasonable to expect some kind of relationship between it and the colloidal index. Fig. 6 shows the thixotropy and colloidal index taken directly from the results of a number of consecutive daily tests. An exact correlation cannot be expected in view of the other factors involved. The principal one of these is the fact that the thixotropy was measured in all stages of chemical treatment, whereas the colloidal index was determined after the optimum dose had been added.

Fig. 7 shows how the general level of the viscosities at the well are dependent on the clay base and the colloidal index. The viscosity in each case was taken by averaging the viscosities recorded by the drillers during the 12 hours before and after the sample was taken. The samples included all types of mud up to about 105 lb./cu. ft. The higher weights cannot be

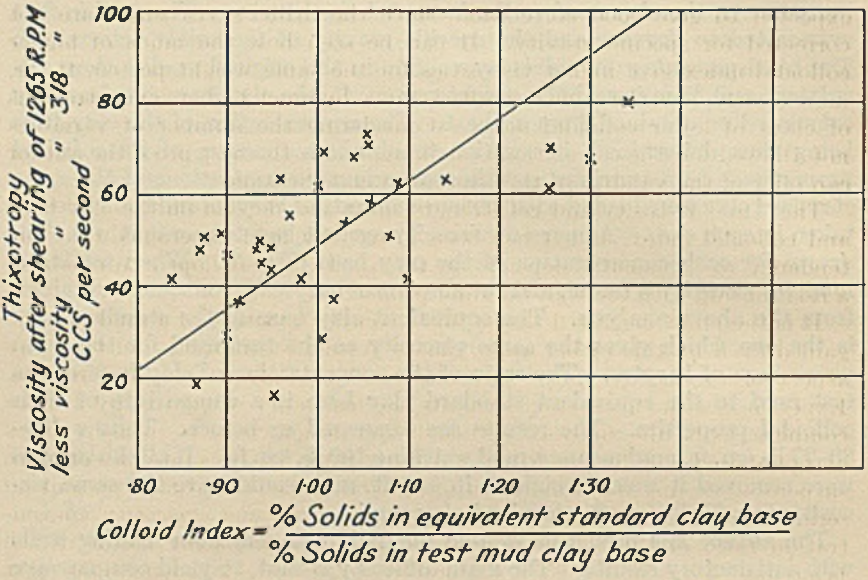

FIG. 6.

GRAPH SHOWING EFFECT OF THE COLLOIDAL PROPERTIES OF THE CLAY BASE ON THE THIXOTROPY OF A MUD.

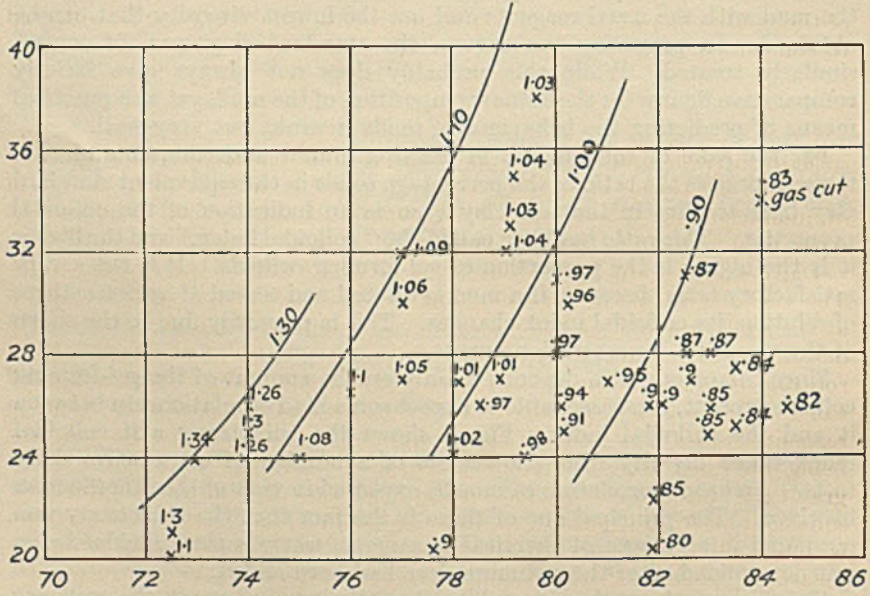

FIG. 7.

GRAPH SHOWING THE EFFECT OF CLAY BASE AND ITS COLLOIDAL PROPERTIES ON THE GENERAL LEVEL OF VISCOSITY AT THE WELL.

Figures on the chart are the colloidal index which is

$$\frac{\text{\% Solids in the equivalent standard clay base}}{\text{\% Solids in the test mud clay base}}.$$

Lines represent muds of equal colloidal index.

expected to give good correlation, since the driller's viscosities are not corrected for specific gravity. It can be seen how the muds of higher colloidal index give higher viscosities for the same weight per cu. ft. or, more clearly, how the same viscosity is given by much higher concentrations of clays of lower colloidal index. Considering the number of variables not allowed for, the correlation is striking, and is the best proof the author can offer of the validity of this system of classification.

The " in " viscosity and gel strength must also depend on the clay base and colloidal index, and it has been proved by field experience that the tendency to " gas-cut " depends on them. It is also hoped to establish a relationship with the wall-building properties.

It is therefore suggested that a fairly complete picture of a mud may be gained even by this rather approximate system of analysis, and that any aspect of the behaviour of the mud may be judged from the results. Experience with this system suggests the feasibility of control by a more complete hydraulic analysis, without using direct viscosity measurements except those taken by the driller. A fairly rapid analysis into two or more suspensoid groups and a more truly colloidal suspension should present no serious practical difficulty. The concentration and viscosity–concentration ratio of this suspension would be the dominating factor in the behaviour of the mud. With moderately dilute colloidal suspensions most of the difficulties of viscosity measurements would be removed. From the experience gained with the present system of analysis it seems likely that the results would be a trustworthy basis on which to judge the performance of the mud at the well, but of course much data would have to be collected to confirm this.

CONTROL.

Many articles and papers appear on various phases of the subject of mud, but the author has not yet come upon one giving details for a system of mud control. Therefore a brief outline of the method used on the Apex field is given for what it is worth.

Daily samples are brought into the laboratory from the drilling wells. The viscosity at speeds corresponding to the " in " and " out " viscosities at the well are taken on the motor-driven paddle viscometer as previously described. The effect of chemical reagents is tried at the fast rate of rotation because the results are obtained much quicker. Quebracho and caustic soda solution, usually in the ratio 1·6 to 1·0, is the most common reagent. Silicate of soda is sometimes used, and good results are now being obtained from compounds containing complex phosphates. The gravity is determined by hydrometer, and a rough figure is obtained for sand content by dilution and settling. A figure for gas entrained in the mud is determined by taking a fixed volume in a special density cup, adding a measured amount of water, shaking up and allowing the gas to escape. The density cup is again filled with the diluted mud and weighed. From the proportion of water added it can easily be calculated what the original weight of the mud would have been had there been no gas in it, and the difference between this and the actual weight is an approximate measure of the amount of gas in the mud. The diluted mud from the density cup is then evaporated rapidly to dryness and again weighed. The loss of weight gives the volume of water and by difference the volume of the solids, and hence their specific gravity. From these results the clay base and the colloidal index are ascertained. All results are recorded and a daily graph is kept of the more important factors, including the results obtained at the well.

The driller is provided with the local I.P.T. viscometer and a mud scale. He takes readings at frequent intervals. Whenever the viscosity rises above a given figure he adds a " dose " to the mud. This is repeated when the viscosity rises again. This " dose " is fixed on the results of the laboratory tests described above and is changed from time to time as conditions change. It usually consists of a stated amount of chemical reagent and water. The water is measured in by means of a simple constant-head meter discharging through interchangeable chokes. The " dose " is added over the period of one circulation.

The " dose " is determined as follows: In normal circumstances the optimum amount of chemical reagent is put in with each dose. This amount is judged from the laboratory test and past experience. If subsequent tests show the amount too much or too little, it is adjusted accordingly. The amount of water depends entirely on the clay base and whether it is desirable to allow it to rise or to fall.

The general aim is to allow the base to rise as much as possible, in order to save barytes. Much more data need to be collected before it can be decided to what limit it is economical to allow the base to rise. It must obviously depend largely on the drilling conditions and the nature of the clay base. At the moment, lower bases are held for muds of high colloidal indices because of the higher " out " viscosities (see Fig. 7) and the greater tendency to gas-cut.

Careful control of the clay base has been found to yield considerable economies in the use of barytes. This is believed to be due to the fact that high viscosities are avoided. As soon as the viscosity rises above a given figure, the driller treats the mud. Clay-base control merely represents a system whereby the minimum amount of water is used to keep within the desired range of viscosities. Low viscosities are economical because with them less solids are picked up and dispersed through the mud and, more important, there is less tendency to gas-cut. (This does not take into account the many advantages of low viscosity to the driller. That viscosity can very materially affect the speed of drilling, especially in deep holes, has been proved several times on the Apex field.) That low viscosities are economical in barytes consumption is shown in Fig. 8, which gives the barytes used to maintain weight in wells before and after the policy was introduced in full on the Apex field.

Blow-outs and Gas-cutting.

The connection between blow-outs and gas-cutting is a point which needs emphasizing. With most blow-outs it can be shown that the reservoir pressure did not exceed that of the hydrostatic head of mud of the gravity that was being maintained, or even that of water. Therefore the blow-out must have been preceded by gas-cutting, without which it would not have occurred. Such gas-cutting can take place merely from the gas in the sand cut by the bit. Also it must be remembered that gas will tend to seep into the mud from sands already penetrated, because of the difference in specific gravity between it and the mud. This will not be controlled by the difference in pressure between the gas and mud column, but by

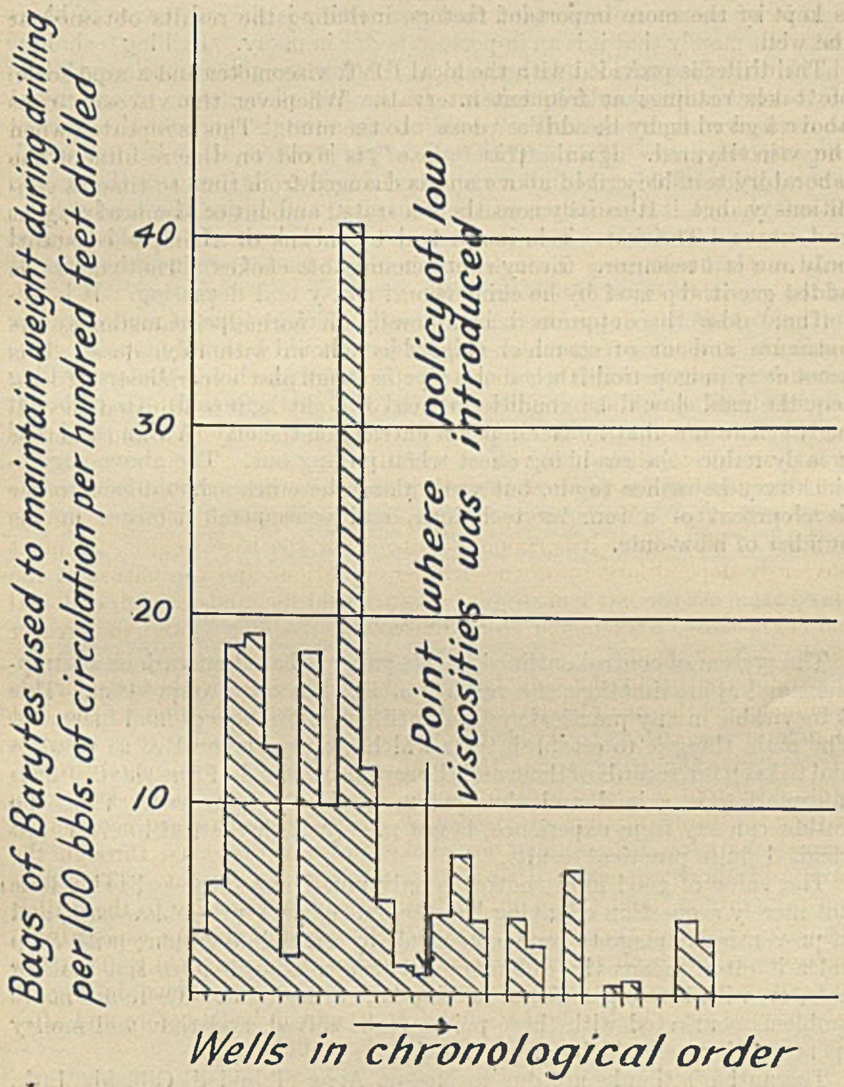

FIG. 8.

DIAGRAMMATIC REPRESENTATION OF THE AMOUNT OF BARYTES USED TO MAINTAIN WEIGHT IN INDIVIDUAL WELLS.

the formation and maintenance of a good mud sheath on the face of the sand. It is significant that blow-outs often occur a long time after the sand has been penetrated, and frequently when out of the hole when conditions for gas-cutting are optimum.

It is by no means contended that gas-cutting is the sole cause of blow-outs, but merely that it is an important factor in many. Drilling technique should therefore take it into account, and below are given some suggestions as to what steps should be taken. In the first place, some means might be devised whereby the driller would obtain a rough figure for the amount of gas in the mud. If a sharp increase of gas is observed, he is forewarned. The addition of barytes is not a very good remedy. It increases the margin of safety, but it also increases the viscosity, and hence the tendency to gas-cut, and a vicious circle is apt to be established. It should be used only as a last resource. Every effort should be made to reduce the amount of the gas in the mud by lowering the viscosity and degassing. It is unfortunate that there appears to be no really efficient type of mud degasser available, and some research on this subject should be amply repaid. The most dangerous period is when the pipe is out of the hole. Before pulling out, the mud should be conditioned and the gas removed. Care should be taken to see that no fresh gas is entering the mud. A thin mud will greatly reduce the swabbing effect when pulling out. The above suggestions may be rather vague, but work along these lines should lead to the development of a sounder technique, and consequent decrease in the number of blow-outs.

CONCLUSIONS.

The system of control outlined in this paper is based on various assumptions and approximations the validity of which is open to question. This is inevitable in any practical system until our knowledge of mud increases. The main thing is to establish tests which are as comparative as possible and to keep full records of them and the results obtained. From these records information is gained and the system gradually improved. This, the author can say from experience, is not merely of theoretical interest, but yields definite practical results.

The value of good mud control is only now being fully realized. It is not merely a question of saving barytes, but of preventing blow-outs, and of preventing damage to producing sands by water infiltration; with deep holes it often means the difference between making hole or not making hole, or, with heaving shale, of making hole or losing it. There are many problems connected with these points to be solved, and time and money spent on them should be amply repaid.

The author's thanks are due to Messrs. Apex (Trinidad) Oilfields, Ltd., for permission to publish this paper, and to Mr. G. H. Scott for frequent help and advice.

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[1] "Evaluation of Rotary Drilling Mud," by P. H. Jones and E. C. Babson, *Oil Weekly*, 2.12.35.

[2] "Drilling Mud," *Trans. Min. and Geol. Inst. of India*, Vol. XXXII.

DISCUSSION.

THE CHAIRMAN (Mr. L. A. BUSHE (Apex)), congratulating Mr. Darley on a very interesting and valuable paper, said that apart from the scientific aspect of mud treatment there were several questions of a practical nature he would like to ask members to deal with during the discussion: How many fields were using the local I.P.T. viscometer as their standard field measure of viscosity? What limits of viscosity were allowed on the field? What percentage of sand was allowed in the muds, and were de-sanders used to any extent? What types of mud degassers were used?

Mr. Bushe also asked speakers to give some indication as to whether they thought it was worth while continuing with these meetings while the war was on, as they would note that very few members of the Institute were present that evening, and the Committee was not sure that it might not perhaps be better to let the local Branch lie dormant until such time as there was not such a pressure of work that it made the obtaining of papers almost an impossibility.

The Chairman then declared Mr. Darley's paper open for discussion.

MR. G. H. SCOTT (Apex) wished to add his congratulations to those of Mr. Bushe. The paper was, to his mind, one of the first that had actually tried to strike a balance between the purely scientific aspect of mud and the practical side. In this work Mr. Darley must be given all credit for having arrived at a basis for mud treatment which, although admittedly purely applicable to local conditions, did correlate the viscosity of the mud as measured by the driller in the field with the laboratory performance.

With reference to the case of cement-cut muds, the I.P.T. viscometer, owing to its rather considerable head, gave a very high rate of shear through the orifice; only a few days ago he had a case where the I.P.T. viscometer was only 22 seconds, but the thixotropy was so high that the mud would not even flow through the laboratory viscometer after shearing.

He would ask the author to enlarge on the importance of the colloidal index when wishing to get Schlumberger plummets to bottom or when running casing.

Mr. Scott referred to the question of variation in the size of hole. If one had changes in the size of the hole there was always the danger, especially in a deep hole, that the large hole above the pilot hole would ultimately build up to the diameter of the pilot hole. He believed Mr. Darley could give some actual cases of this.

As the author had pointed out, his use of the term "suspensoid" was perhaps a little unfortunate; "suspensoid" as used in pure colloidal chemistry was rather limited to the hydrophobic type of sol. He could think of no other term for particles which, although larger than colloidal dimensions, were held in suspension by the nature of the dispersive base and by their concentration.

With regard to the question of gas-cutting and blow-outs, Mr. Scott pointed out that the modern tendency to "slim" holes tended to aggravate the danger unless rigid mud control was observed. In many cases the interstitial gas in the sand actually excavated by the bit was sufficient to cause extensive gas-cutting. Furthermore, the tendency to swab a hole when pulling out was increased as hole diameters decreased, and it was of prime importance to condition the mud and secure a low viscosity prior to a "round trip."

MR. DARLEY, replying, said that with regard to the colloidal index when running Schlumberger plummets, it was a point that had to be taken very much into account. If you had a mud yielding quite a good viscosity at the well but having a high colloidal index, it was going to gel fast, and by the time the Schlumberger plummet got down it might be thick enough to prevent it reaching bottom. He had not done many experiments in this direction, but generally found that agents such as quebracho would reduce viscosity but did not greatly affect the rate at which the mud gelled. The only solution, he thought, was to dilute the mud down.

On the point of different sizes of hole, he thought perhaps the worst case was when cavities due to caving were obtained, resulting in changes of velocity which caused the deposition of cuttings.

Mr. G. S. Taitt (United British Oilfields of Trinidad) said he was interested in the Chairman's remarks with regard to the holding of meetings of the Branch. This was the first time he had had the opportunity of attending one himself, but he felt it would be a great pity if the meetings had to be given up, though he quite realized the difficulties under present conditions. He suggested it would be better to have an occasional paper—say once in one or two months—rather than close down the Branch altogether, because there was always a certain amount of initiative available.

He thought Mr. Darley was to be congratulated on the systematic way in which he had dealt with his particular mud problems.

In the first part of the paper, dealing with flow characteristics of mud, several references were made to the possibility of discounting the effect of thixotropy—*e.g.*, in measuring viscosities after an interval of not more than half a minute. However, the speaker had had some experience of muds gelling almost instantaneously, which had prevented the obtaining of accurate viscosities and comparative figures, so that he felt it could not be completely neglected. He did not know if Mr. Darley had come across such conditions.

He thought the importance of thixotropy had not perhaps been stressed enough in the paper, because even with mud which showed a very good condition while circulating, its thixotropy was of great importance; for example, if the pumps were shut down, even for a short time, when starting up again high initial pressure due to gelling might cause complete loss of circulation.

On the question of the colloidal index, tests might be made by preparing standard muds; but had Mr. Darley noticed the change which took place after a period of circulation with a given mud? They had observed that with some muds the clay content appeared quite satisfactory at the beginning, but after a while, possibly due to crushing or grinding of clay particles, the colloidal content appeared to have gone up.

In connection with the degassing of muds, had Mr. Darley had experience of vacuum degassifying?

With regard to plastering, Mr. Darley had stated in his paper: "Also it must be remembered that gas will tend to seep into the mud from sands already penetrated because of the difference in specific gravity between it and the mud. This will not be controlled by the difference in pressure between the gas and mud column but by the formation and maintenance of a good mud sheath on the face of the sand."

Some experiments had been made which tended to suggest that the pressure required to deplaster was very low indeed with a good mud, and therefore he wondered why Mr. Darley attached such importance to the nature of the mud sheath for holding back gas.

Mr. Darley, replying on the question of muds setting instantly due to thixotropy, said that he had, of course, had experience of such a condition. His actual remark had been made in connection with his tests of the rate of discharge against head. He was merely trying to establish the relationship and get some values for the Apex field. The muds used were all fairly thin muds, gelling effect was slight, and the actual motion through the viscometer was quite sufficient to prevent the thixotropy from having any effect. He quite agreed that with very thick muds of high thixotropy, viscometer readings were apt to be very seriously upset.

The change in viscosity due to circulation was very readily understandable. The point was well illustrated by the difficulty in obtaining a constant viscosity when making up a mud from the raw clay. His own standard muds were mixed by placing in a concrete mixer and rotating for a day, and even then, on putting them in the viscometer and rotating he could still get a rise in viscosity over a period of time. This was undoubtedly due to the particles of the clay becoming more and more dispersed and becoming finer; their surface area was increased and colloidal properties came more and more into play.

With regard to vacuum degassing, he had no field experience, but had tried it in a small way in the laboratory, just holding a small vacuum of a few inches. The difference he had obtained was practically negligible.

With regard to plastering, his answer to the question whether gas could seep into the mud against a higher head if there was not a mud sheath, was emphatically yes. Gas would tend to rise purely because it was lighter than the mud. If one had a closed glass tube with mud in it and an air space at the top, and the tube was reversed end for end, the air bubble would go back to the top. The function of the mud sheath was that of a membrane to prevent that action. He had done no experiments on the force necessary to remove the mud sheath, but this must obviously be dependent on the difference between the reservoir pressure and the pressure of the mud column. That was where loading of the mud was of value.

Mr. Taitt explained that he realized the necessity for some mud sheath (which, he assumed, would be present in any case), but he wondered if Mr. Darley had noticed any difference with muds of different plastering qualities.

Mr. Darley replied that he had no comparisons from which conclusions could be reached.

Mr. N. Healey (United British Oilfields of Trinidad), speaking on the question of gas entering mud, said that when a sand had been drilled, if the hydrostatic head of the mud was higher than the reservoir pressure, laboratory experience showed that the absorptive effect would give rise to gas-cutting, even though there was a far greater pressure due to the mud than to the reservoir.

He also raised the point about the abrasiveness of particles in mud and particle size. He believed particle sizes had very little to do with abrasive action and it was much more due to particle shape. If you had sand particles which were perfectly rounded, there would be very little abrasive action indeed, but if they had sharp corners, even if of very small size, they would have considerable abrasive action. Laboratory experiments could be done with particles of different kinds.

Mr. Darley agreed that shape must have a very great deal to do with it, but, given the same shaped particles, the larger particles would give deeper scratches, smaller ones having more of a polishing effect.

Mr. Healey suggested, with regard to the colloidal index, that if a standard mud could be prepared—which was admittedly difficult—and some amount of standard clay and water put in and ground for some definite time, taking some standard clay such as Aquagel instead of local Trinidad clay, then perhaps one could get an almost universal standard mud, which would help in comparing muds in different countries.

Mr. Darley thought that this would be a very useful thing for comparing results between one field and another.

Mr. Scott remarked that bentonites were so liable to variation that he did not think Aquagel could be trusted any more than clay as a standard.

Mr. Darley stated that he had had samples of bentonites which had completely lost their gelling qualities merely with age. Therefore he did not think such a sensitive clay would be suitable for preparing a standard mud.

Mr. Healey mentioned the difficulty in correlating results on the rig with those obtained in the laboratory. With regard to viscosity, an even better criterion would be to take the pump pressure and rate of circulation. If there was streamline flow in the annulus, the laws of viscosity should apply, and from that it should be possible to get some idea of the viscosity. It would then remain to correlate this with the laboratory viscosity.

Mr. Darley pointed out that the trouble would be to standardize conditions. There were so many other important factors—*e.g.*, if a change was made from 4-inch to 2-inch drilling pipe, the pump pressures would almost double themselves.

Dr. A. J. Goodman (Cory Bros.) mentioned a mud degasser which he had seen in the *Oil Weekly* about two months previously.

Mr. Darley said he had recently come across one which had a vacuum at the top and baffles to increase the area exposed to the vacuum. He had not used anything similar to that.

Mr. Bushe said that the Committee hoped to have another paper late in June. If any member had a particular urge to read a paper before the Branch and would write to the Secretary, they would see what could be done about it.

In conclusion, he proposed a vote of thanks to Mr. Darley for his paper, and also to the Apex Club for the use of their Clubroom.

SYNTHESIS AND PROPERTIES OF MONO-NORMAL-ALKYLBENZENES.*

PART I.—METHODS FOR THE ALKYLATION OF BENZENE.

By G. Shen, M.Sc., A.M.Inst.Pet., T. Y. Ju, B.Sc., Stud.Inst.Pet., and C. E. Wood, M.Sc., A.I.C., F.Inst.Pet.

Synopsis.

The principal available methods for preparing alkyl (long-straight chain) benzenes are reviewed critically. Seven methods are considered in detail—namely, (1) Fittig's synthesis; (2) condensation of alkyl halides with benzene; (3) condensation of olefines with benzene; (4) condensation of alcohols with benzene; (5) interaction of inorganic and organic esters with benzene; (6) Grignard synthesis; and (7) hydrogenation of alkyl aryl ketones. Each reaction is considered and explained in electronic terms. Certain complicated reactions difficult of explanation on a logical basis and which lead to contradictory conclusions can be explained by, and are in agreement with, the fundamental ideas of activation and addition of an addend after cleavage in accordance with Markownikoff's rule. The first six methods are considered unsuited with regard to good yield and purity of the product.

The hydrogenation of alkyl aryl ketones prepared by the Friedel and Crafts' reaction (acylation of benzene) appears to be the best method for obtaining the large quantities of hydrocarbons which are necessary for the determination of cetane numbers. This process is on the whole of a simple character, not involving rearrangement of alkyl groups, and is unaccompanied by side reactions; further, the process does not involve a too-elaborate organic technique. Palladium is the best catalyst for carrying out the hydrogenation. Considering different chemical methods of reduction, the Clemmensen's method is to be preferred.

Numerous methods are available for the preparation of the mono-*n*-alkylbenzenes, and in a survey of the literature certain methods are difficult, others easy in technique. The yield of the product is, as a rule, low, and the pure hydrocarbon can be isolated only with difficulty. Thus, a comparative and critical study of all available methods mentioned in the literature has been made. Eight principal methods for the alkylation of the aromatic nucleus are in use :—

(1) Fittig's synthesis.

(2) Condensation of alkyl halides with benzene using aluminium chloride (Friedel and Crafts' reaction) or hydrogen fluoride.

(3) Condensation of olefines with benzene in the presence of hydrogen fluoride, sulphuric acid, aluminium chloride, ferric chloride, or phosphoric acid.

(4) Condensation of alcohols with benzene in the presence of boron trifluoride, aluminium chloride, phosphorus pentoxide, or boric acid (through the intermediate formation of R_3BO_3 from the corresponding alcohol, ROH, and boric acid) and aluminium chloride.

(5) Interaction of inorganic and organic esters with benzene in the presence of boron trifluoride, aluminium chloride, or hydrogen fluoride.

(6) Grignard synthesis.

(7) Hydrogenation of alkyl aryl ketones which may be prepared by the following principal methods :—

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* Paper received 8th May, 1940.

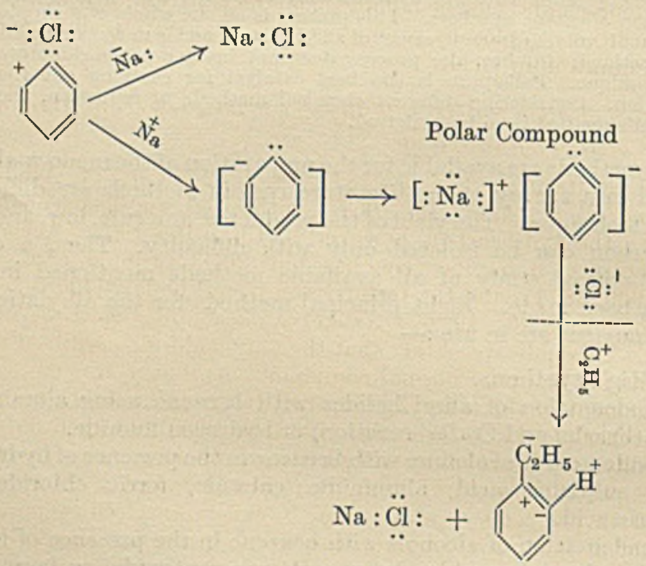
(*a*) Acylation of benzene by Friedel and Crafts' reaction.
(*b*) Grignard synthesis.
(*c*) Passing the vapours of benzoic and the fatty acids through heated manganous oxide or thorium oxide according to the elegant method of Sabatier.

(8) Reaction between *cyclo*propane and benzene using as catalyst hydrogen fluoride. The explanation of the reaction possibly involves an ionic mechanism (Simons, Archer, and Adams, *J. Amer. chem. Soc.*, 1938, 60, 2955–2956).

It is interesting to review briefly the methods which have at times been utilized for this particular type of alkylation.

(1) *Fittig's Synthesis.*

The first stage in the accepted explanation of the Fittig reaction (Tollens and Fittig, *Annalen*, 1864, 131, 303) is the formation of sodium phenyl, C_6H_5Na (Acree, *Amer. Chem. J.*, 1903, 29, 588; Schlubach, *Ber.*, 1922, 55, 2889), on the immediate liberation of the phenyl radical. The second stage involves the reaction of sodium phenyl with the alkyl or aryl halide in accordance with the following explanation, in which it is interesting to note the amphoteric character of carbon :—



The polar compound reacts more readily with the alkyl halide than with the aryl halide, and on this account the yield of alkylated benzene is much better than would be anticipated in view of the three possible products. Butylene, butane (both formed from the free radicals), and diphenyl are by-products. Sodium is the metal most useful for coupling alkyl and aryl radicals. Bachmann and Clarke (*J. Amer. chem. Soc.*, 1927, 49, 2089) found that the action of sodium on chlorobenzene at the boiling point gave benzene, diphenyl, *o*-diphenylbenzene, triphenylene, 2 : 2-diphenyl-

diphenyl and resinous tars; further, the action of sodium on *n*-heptyl bromide gave *n*-heptane, heptylene, tetradecane, heneicosane, and high-molecular-weight hydrocarbons. Copper, however, is most suited for this type of coupling.

n-Amylbenzene was prepared by Schramm (*Annalen*, 1883, 218, 388) by action of sodium on a mixture of benzyl bromide and butyl bromide. The yield was about 25 per cent. theoretical, dibenzyl and *n*-octane and some unidentified hydrocarbons being formed at the same time. Radcliffe and Simpkin (*J. Soc. chem. Ind.*, 1921, 40, 119–122T) use benzyl chloride instead of benzyl bromide, and the yield is similar to that obtained by using benzyl bromide, but requires a longer time for completion of the reaction. *n*-Amylbenzene was also prepared by Radcliffe and Simpkin (*loc. cit.*) by treating a mixture of phenyl bromide and amyl bromide with metallic sodium, the yield being two to three times greater.

Possible combinations of organo-sodium compounds and organic chlorides in the Fittig's synthesis of *n*-amylbenzene have been studied recently by Morton and Fallwell (*J. Amer. chem. Soc.*, 1938, 60, 1429–1431). The yield was low (5–11 per cent.). However, a better yield (54–70 per cent.) was obtained by using sodium benzyl instead of sodium phenyl.

In view of these side reactions, which, in certain circumstances, might well overwhelm the required synthesis, the Fittig reaction is not satisfactory for preparing long-chain benzene homologues, the isolation of which in the pure condition from complex by-products entails elaborate technique with consequent diminution in yield.

(2) *Condensation of Alkyl Halides with Benzene in the Presence of Aluminium Chloride or Hydrogen Fluoride.*

Friedel and Crafts (*Compt. rend.*, 1877, 84, 1392, 1450; cf. Ashdown, *Industr. Engng Chem.*, 1927, 19, 1063) synthesized amylbenzene using amyl chloride; the hydrocarbon, however, had b.p. 185–190°. According to recent literature, *n*-amylbenzene has b.p. 205·3° (Simon, *Bull. Soc. chim. Belg.*, 1929, 38, 47) and the tertiary compound boils at 190° (Verley, *Bull. Soc. chim.*, 1898, (3), 19, 67; Tzukervanik, *J. Gen. chem. U.S.S.R.*, 1935, 5, 117). This indicates either that the amyl chloride used by Friedel and Crafts was not the pure normal compound, or, if such was employed, then rearrangement of the *n*-amyl group had taken place in the reaction. Simon and Archer (*J. Amer. chem. Soc.*, 1938, 60, 2953) obtained only fair yields of the alkylated benzenes by using hydrogen fluoride. From *n*-propyl bromide a 48 per cent. yield of propylbenzenes (containing 88 per cent. *iso*propyl and 12 per cent. *n*-propyl) and from *tert.*-amyl chloride a 41·5 per cent. yield of mono-*tert.*-amylbenzene were obtained. The following shows the ease of rearrangement of the halides :—

Temperature.	Halide.	Product.
—	*n*-Propyl halide	*iso*Propylbenzene predominates.
0°	*n*-Butyl halide	*n*-Butyl- and *sec.*-butyl benzene.
Higher temperature than 0°	*n*-Butyl halide	*sec.*-Butylbenzene.
As low as −18°	*iso*Butyl halide	*tert.*-Butylbenzene exclusively.
—	*iso*Amyl halide	*iso*Amylbenzene and *tert.*-amylbenzene.

The reaction is considered below in terms of electronic structures :—

$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2Cl + AlCl_3 \longrightarrow [CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2\overset{+}{Cl} - Al\overset{-}{Cl}_3]$$

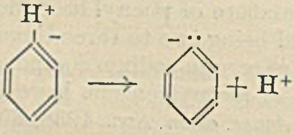
This reaction is catalytic—*i.e.*, no stable organo-aluminium compound is formed :—

$$[CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2\overset{+}{Cl} - Al\overset{-}{Cl}_3] \longrightarrow CH_3{\cdot}CH_2{\cdot}CH = CH_2 + HCl + AlCl_3$$

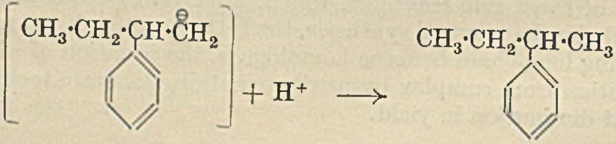
Activation of unsaturated hydrocarbon :—

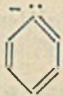
$$CH_3{\cdot}CH_2{\cdot}CH = CH_2 \longrightarrow CH_3{\cdot}CH_2{\cdot}\overset{\oplus}{C}H - \overset{\ominus}{C}H_2$$

Dissociation of proton from benzene ring :—



Then addition of the phenyl radical to the activated hydrocarbon to give the *iso*alkylated benzene after proton addition :—



The addition of proton H+ and phenyl  to the unsaturated hydrocarbon is in accordance with Markownikoff's rule (*Annalen*, 1870, 153, 256; *Compt. rend.*, 1875, 81, 670). The reaction *is not* the breaking down of an activated covalent alkyl halide into ions

$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}Cl \longrightarrow CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2^+ + Cl^-$$

then dissociation of a proton from the benzene nucleus and addition of the alkyl radical to the residual phenyl grouping, this would lead to the formation of a *normal*-alkylated benzene.

The cleavage of the halide has been investigated by H. Gilman and R. R. Burtner (*J. Amer. chem. Soc.*, 1935, 57, 909) in the furan series, the *tert*.-butyl derivative having been obtained from each of the following: *n*-hexyl bromide, *n*-octadecyl bromide, and *n*-amyl chloride.

The control of conditions for introducing an alkyl group is easy, but there is possible rearrangement and cleavage of the *n*-alkyl group.

The use of *n*-alkyl halide (particularly of higher *n*-halides) for the preparation of mono-*n*-alkylated benzenes was therefore not practicable.

It is to be noted that Norris and Rubinstein (*J. Amer. chem. Soc.*, 1939, 61, 1163–1170) obtained *s*.-triethylbenzene in 85–90 per cent. of the theoretical from ethylation of benzene with ethyl chloride and ethyl bromide when the molecular ratio of aluminium chloride to benzene was 1 : 1. The methylation of benzene with 3 moles of methyl halide gives 1 : 2 : 4-trimethylbenzene at 0° and 1 : 3 : 5-trimethylbenzene at 100°.

(3) *Condensation of Olefines with Benzene.*

The condensation of olefines with benzene in the presence of various condensing agents gives the same products as those obtained from the corresponding alkyl halides.

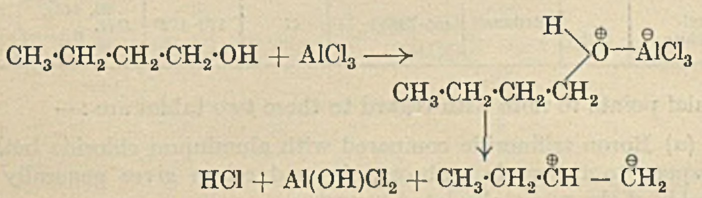
Olefine.	Condensing agent.	Product.	Reference.
Propene	$AlCl_3$	*iso*Propylbenzene	Berry and Reid (*J. Amer. chem. Soc.*, 1927, 49, 3142).
,,	BF_3	,,	Slanina, Sowa, and Nieuwland (*ibid.*, 1935, 57, 1547).
n-Butene	BF_3	*mono*Butylbenzene isomers	*Ibid.*
Propene	HF	84% yield *iso*propylbenzene	Simon and Archer (*ibid.*, 1938, 60, 2952).
*iso*Butene	HF	44% yield mono-*tert.*-butylbenzene and poly-alkylated benzenes	*Ibid.*
,,	96% H_2SO_4	77% yield *p*-di-*tert.*-butylbenzene and small percentage of mono- and tri-derivatives	Ipatieff, Corson, and Pines (*ibid.*, 1936, 58, 919).
*iso*Pentene	96% H_2SO_4	20% *tert.*-amylbenzene 56% diamylbenzene	Ipatieff, Pines, and Schmerling (*ibid.*, 1938, 60, 353).
tert.-Pentene	96% H_2SO_4	18% *tert.*-amylbenzene 50% di-*tert.*-amylbenzene	*Ibid.*
Propene	$FeCl_3$	91% *iso*propylbenzene	Potts and Carpenter (*ibid.*, 1939, 61, 663).
*iso*Butene	$FeCl_3$	89% *tert.*-butylbenzene	*Ibid.*
,,	P_2O_5	*tert.*-Butylbenzene	Malishev (*ibid.*, 1935, 57, 883).
Olefines from cracked gasoline and kerosine.	$AlCl_3$	Mono-alkylated benzenes (structure of alkyl groups apparently not investigated)	Tilicheev and Kuryndin (*Neft. Khoz.*, 1930, 19, 586).
Olefines (sources as above)	$FeCl_3$	68% yield mono-alkylated benzenes (alkyl groups not normal)	Kuryndin, Vocvodova, and Rasskazova (*J. appl. Chem.* (*U.S.S.R.*), 1937, 10, 877).

Ipatieff, Pines, and Komarewsky (*Industr. Engng Chem.*, 1936, 28, 222) have used phosphoric acid as condensing agent.

In this case (3), also, the addition of benzene to the olefine takes place according to Markownikoff's rule. Similar remarks as made under (2) apply here. The preparation of *n*-alkylated benzenes in reasonable yield under investigated conditions is impossible.

(4) *Condensation of Alcohols with Benzene.*

In the condensation of alcohols with benzene (in the presence of dehydrating agents as BF_3, $AlCl_3$, P_2O_5, H_2SO_4, etc.) *sec.*- and *tert.*-alkylbenzenes are obtained. The reaction proceeds with the intermediate formation of the olefine (cf. Tzukervanik, *J. Gen. Chem.*, *U.S.S.R.*, 1935, 5, 117) and then addition of benzene as explained above. Taking *n*-butyl alcohol as an instance, the first two stages of the reaction may be represented thus :—



$$CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}OH + AlCl_3 \longrightarrow \begin{matrix} H \diagdown \; \overset{\oplus}{} \; \overset{\ominus}{} \\ \rangle O{-}AlCl_3 \\ CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2 \diagup \end{matrix}$$

$$\downarrow$$

$$HCl + Al(OH)Cl_2 + CH_3{\cdot}CH_2{\cdot}\overset{\oplus}{C}H - \overset{\ominus}{C}H_2$$

The following table shows the products of alkylation.

Alcohol.	Condensing agent.	Product.	Reference.
n-Propyl	BF_3	*iso*Propylbenzene	McKenna and Sowa (*J. Amer. chem. Soc.*, 1937, 59, 470).
*iso*Propyl	BF_3	"	
n-Butyl	BF_3	*sec.*-Butylbenzene	
sec.-Butyl	BF_3	"	
*iso*Butyl	BF_3	*tert.*-Butylbenzene	
tert.-Butyl	BF_3	"	
*iso*Propyl	80% H_2SO_4	*iso*Propylbenzene and poly-substitutes	Meyer and Bernhauer (*Monatsh.*, 1929, 53–54, 721).
n-Butyl	H_2SO_4	*sec.*-Butylbenzene and di-substitutes	*Ibid.*
*iso*Butyl	H_2SO_4	*tert.*-Butylbenzene and di-substitutes	*Ibid.*
tert.-Butyl	H_2SO_4	*tert.*-Butylbenzene and di-substitutes	*Ibid.*
Methyl	$AlCl_3$	21% yield toluene	Norris and Sturgis (*J. Amer. chem. Soc.*, 1939, 61, 1413).
Ethyl	$AlCl_3$	50% yield ethylbenzene	*Ibid.*
*iso*Propyl	$AlCl_3$	*iso*Propylbenzene	Huston and Hsieh (*ibid.*, 1936, 58, 439).
"	$AlCl_3$	*iso*Propylbenzene and ethylbenzene	Norris and Sturgis (*loc. cit.*).
tert.-Butyl	$AlCl_3$	84% *tert.*-butylbenzene	*Ibid.*
"	$AlCl_3$	67% *tert.*-butylbenzene	Huston and Hsieh (*loc. cit.*).

The disadvantages of using alcohols are similar to those of using olefines or alkyl halides. In reactions (2) and (3) the $AlCl_3$ or other condensing reagent employed reacts catalytically; in (4), however, $AlCl_3$ (P_2O_5, H_2SO_4, etc.) dehydrates and then the excess $AlCl_3$ acts as a powerful catalyst.

(5) *Interaction of Inorganic and Organic Esters with Benzene.*

The reagents used to promote the condensations are BF_3, HF, and $AlCl_3$. Some recent results are tabulated below.

TABLE I.

Ester inorganic.	Condensing agent.	Temp. (highest), ° C.	Nature of alkyl group in benzene.	% yield of mono-alkylated benzenes.	B.p. of mono-alkylated benzenes. ° C.	Reference.
1. *n*-Propyl-sulphate	BF_3	Refluxed	*iso*Propyl	45	—	McKenna and Sowa (*J. Amer. chem. Soc.*, 1937, 59, 1204).
2. *iso*Propyl-sulphate	"	"	"	38	—	*Ibid.*
3. *n*-Butyl-phosphate	"	"	*sec.*-Butyl	11	—	*Ibid.*
4. Diethyl-sulphate	$AlCl_3$	70°	Ethyl	71·4	133–137°	Kane and Lowy (*J. Amer. chem. Soc.*, 1936, 58, 2605).
5. Dibutyl-sulphate	"	70°	Isomeric mixtures of butyl benzenes	43·6	165–175°	*Ibid.*
6. *n*-Propyl-sulphite	"	60°	*n*-Propyl	66	150–151°	Everett Bowden (*J. Amer. chem. Soc.*, 1938, 60, 645).
7. *n*-Butyl-sulphite	"	Refluxed	*sec.*-Butyl	41	170–172°	*Ibid.*

The chief points to note with regard to these two tables are :—

(*a*) Boron trifluoride compared with aluminium chloride both with respect to inorganic and organic acid esters gives generally lower yields of the mono-alkylated benzenes.

(*b*) Using BF_3 (1) the *iso*- or *sec.*-alkylated benzenes are obtained from the *n*-alkyl ester (cf. *n*-propyl and *n*-butyl esters); (2) the mono-*tert.*-alkylated benzenes are obtained from the *iso*alkyl ester.

(*c*) Aluminium chloride gives *normal* and not *iso*propylbenzene under the standardized conditions of the authors mentioned.

TABLE II.

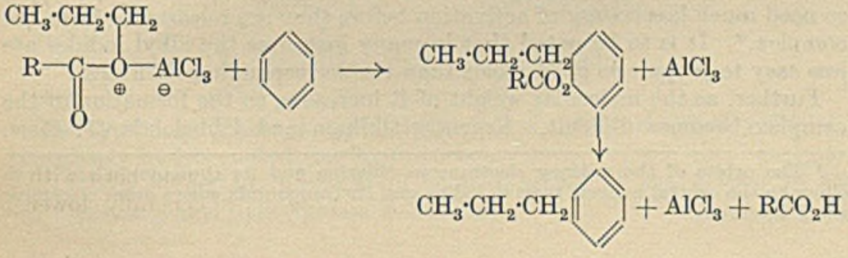
Ester organic.	Con-densing agent.	Temp. (highest), ° C.	Nature of alkyl group in benzene.	% yield of mono-alkylated benzenes.	B.p. of mono-alkylated benzenes, ° C.	Reference.
1. *n*-Propyl formate	BF_3	Refluxed	*iso*Propyl	33·4	—	McKenna and Sowa (*J. Amer. chem. Soc.*, 1937, 59, 1204).
2. *iso*Propyl acetate	,,	,,	,,	10·4	—	*Ibid.*
3. *n*-Butyl formate	,,	,,	*sec.*-Butyl	33·2	—	*Ibid.*
4. *iso*Butyl formate	,,	,,	*tert.*-Butyl	30·6	—	*Ibid.*
5. *sec.*-Butyl formate	,,	,,	*sec.*-Butyl	26·0	—	*Ibid.*
6. *sec.*-Butyl acetate	,,	,,	,,	29·6	—	*Ibid.*
7. *n*-Propyl formate	$AlCl_3$	60°	*n*-Propyl	66	150–151°	Everett Bowden (*J. Amer. chem. Soc.*, 1938, 60, 645).
8. *n*-Butyl formate	,,	75°	*sec.*-Butyl	73	170–172°	*Ibid.*
9. *iso*Propyl acetate	,,	Refluxed	*iso*Propyl	68	145–148°	*Ibid.*
10. *iso*Butyl acetate	,,	60°	*tert.*-Butyl	33	168·5°	*Ibid.*
11. *iso*Propyl acetate	HF	80–100°	*iso*Propyl	53	149–150/742 mm.	Simons, Archer, and Randall (*J. Amer. chem. Soc.*, 1939, 61, 1821–1822).
12. *n*-Butyl acetate	,,	80–100°	*sec.*-Butyl	60	170·5–171/742 mm.	*Ibid.*
13. *sec.*-Butyl *iso*butyrate	,,	80–100°	,,	56	170–171/742 mm.	*Ibid.*
14. *tert.*-Butyl acetate	,,	80–100°	*tert.*-Butyl	71	170–171/742 mm.	*Ibid.*

(*d*) Aluminium chloride reacts in the usual manner to give *sec.*- and *tert.*-butylbenzenes from *n*- and *iso*-butyl esters, respectively.

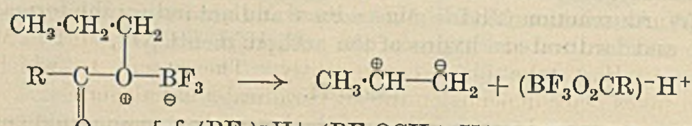
(*e*) Hydrofluoric acid acts similarly to boron fluoride.

(*f*) No rearrangement takes place on using either BF_3 or $AlCl_3$ with regard to the *iso*propyl radical or on using BF_3 with regard to the *sec.*-butyl radical.

A mechanism similar to the Friedel and Crafts' reaction may be postulated for these esters on using aluminium chloride. Co-ordination with the oxygen of the ester grouping occurs, followed by addition of the alkyl radical and the residual complex at the double bond of the aromatic ring, then the elimination of the acid.

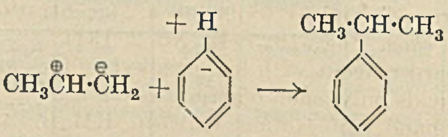


which virtually amounts to the addition of the ions activated $C_3H_7^+$ and $R{\cdot}CO_2^-$ at the double bond. Co-ordination of BF_3 results apparently in olefine formation :—



[cf. $(BF_4)^-H^+$, $(BF_3OCH_3)^-H^+$, $(AlCl_4)^-(RCO)^+$]

then prototropic change and addition take place :—



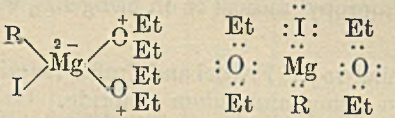
In the case of (*a*), the normal butyl esters and $AlCl_3$ or BF_3, the intermediate is activated butene-1 ($CH_3–CH_2\overset{+}{C}H–\overset{-}{C}H_2$), which forms *sec.*-butylbenzene; (*b*), the *sec.*-butyl esters and BF_3, the intermediate is activated butene-2 ($CH_3{\cdot}\overset{+}{C}H–\overset{-}{C}H{\cdot}CH_3$), which forms *sec.*-butylbenzene; (*c*), the *iso*butyl esters and $AlCl_3$ or BF_3, the intermediate is activated methylpropene $\left(\begin{smallmatrix}CH_3\\CH_3\end{smallmatrix}{>}\overset{+}{C}–\overset{-}{C}H_2\right)$, which forms *tert.*-butylbenzene.

In all cases the hydrogen (Proton) or "positive" part of the benzene nucleus adds to the end carbon atom ($–CH_2$ or $–CH–$) (Markownikoff's rule).

So far as is known, no normal alkylated benzenes (higher than propyl) can be formed by methods (2), (3), (4), and (5), although the known reversal of Markownikoff's rule (addition of haloid acid to olefines in the presence of peroxides) would indicate the possibility of their formation under modified conditions.

(6) *The Grignard Synthesis.*

The formation of the co-ordination complex



proceeds more easily with alkyl or aryl *iodides* than with other halides, for the valency electrons of the iodide ion move under the action of much weaker fields than the valency electrons of the chloride or fluoride ion, and so need much less energy of activation before they are released to form the complex.* It is to be noted that in many instances the alkyl iodides are less easy to prepare in good yields than the corresponding chlorides.

Further, as the molecular weight of R increases, so the formation of the complex becomes difficult. Recently Oldham and Ubbelohde (*J. chem.*

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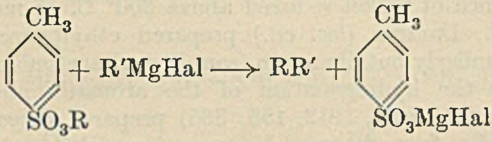
* The orbits of the valency electrons in chlorine and its compounds are much closer to the central nucleus than in iodine and its compounds where other electronic shells intervene.

Soc., 1938, 201) investigated the products of the reaction of various higher-molecular-weight halides on magnesium in the presence of ether. They showed that the three reactions (*a*) formation of the Grignard complex, (*b*) the Wurtz reaction ($2RI + Mg = R_2 + MgI_2$), and (*c*) the formation of olefine and saturated hydrocarbon ($C_{12}H_{25}I + C_{12}H_{25}MgI = MgI_2 + C_{12}H_{24} + C_{12}H_{26}$) take place concurrently. The extent to which each reaction takes place depends on the alkyl radical and halogen.

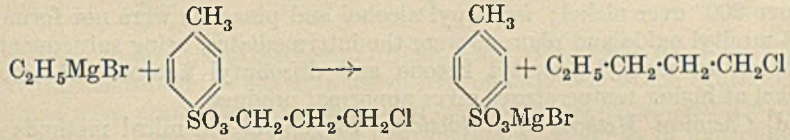
Even considering the preparation of the lower alkyl magnesium complex, C_2H_5MgBr, efficient cooling and stirring are essential to prevent the side reaction: $2C_2H_5Br + Mg \longrightarrow C_2H_4 + C_2H_6$, which is favoured by rapid addition of the bromide and by too small a concentration of ether (Wood and Scarf, *J. Soc. chem. Ind.*, 1923, 42, No. 2, 13T).

Thus the Grignard reagent with higher alkyl halides gives higher hydrocarbons in poor yields only, such reactions being used as a last resort.

On the other hand, interaction between RMgBr and C_6H_5Br gives diphenyl if the reaction goes too violently. Considering the following reaction, $RHal + R'MgHal \longrightarrow MgHal_2 + RR'$ the yields are poor, particularly with tertiary halides. A modification which gives better yields involves the use of aromatic sulphonic acid alkyl esters :—



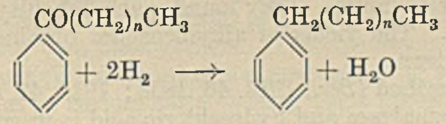
It should be mentioned, however, with regard to the use of alkyl halides in other types of synthesis, that the chain of an alkyl halide may be lengthened by three carbon atoms by treating its Grignard reagent with γ-chloropropyl-*p*-toluenesulphonate :—



This alkyl halide can now be used in hydrocarbon synthesis.

(7) *Hydrogenation of Alkyl Aryl Ketones.*

The controlling of the hydrogenation of the ketone to the second stage —viz., the aryl hydrocarbon—is one requiring considerable experience of hydrogenation technique :—



The first stage yields the secondary alcohol, whilst the final yields the completely hydrogenated ring hydrocarbon.

A study of certain methods of hydrogenation of the ketone to the corresponding aryl hydrocarbon was made.

(a) *Palladium as Catalyst.*—Hartung and Crossley (*J. Amer. chem. Soc.*, 1934, **56**, 158) reduced ethyl phenyl ketone in alcoholic solution to propylbenzene, using palladinized animal charcoal which was prepared according to the direction of Otto and Schroeter (*Ber.*, 1927, **60**, 633). The method was claimed to be successful for the complete intermediate reduction of ketone to hydrocarbon without the formation of secondary alcohol or alkyl*cyclo*hexane.

(b) *Platinum-black as Catalyst.*—Vavon (*Compt. rend.*, 1912, **155**, 286) hydrogenated acetophenone in 60 per cent. aqueous alcohol, giving almost pure methylphenylcarbinol. In ether, alcohol (absolute), ethyl acetate, or acetic acid, however, the product was ethyl*cyclo*hexane. So the control of the second hydrogenation stage is rendered very difficult.

(c) *Nickel as Catalyst.*—The activity of catalytic nickel depends on the method of preparation. The temperature used for reduction of the oxide is an important factor. Darzens (*Compt. rend.*, 1904, **139**, 869) and Brunel (*Ann. Chim. Phys.*, 1905, **6**, (8), 205) found that the higher the temperature of reduction the feebler was the activity of the resulting nickel; hence hydrogenation of the benzene nucleus is slow with catalyst prepared above 300° C.

In the presence of nickel reduced above 300° C. of moderate activity at 190–195° C., Darzens (*loc. cit.*) prepared ethylbenzene from acetophenone and similarly butylbenzene from benzyl acetone. A more active catalyst causes the hydrogenation of the aromatic nucleus. Sabatier and Murat (*Compt. rend.*, 1912, **155**, 385) prepared di*cyclo*hexylpropane, $C_6H_{11}(CH_2)_3C_6H_{11}$, from dibenzyl ketone using nickel at 175°. At higher temperatures—*i.e.*, at 250–300° C.—Haller and Lassieur (*Compt. rend.*, 1910, **150**, 1013) hydrogenated methyl nonyl ketone using nickel, and obtained nonane, C_9H_{20}, a pinacone, $C_9H_{19}C(CH_3)_2{,}CO{\cdot}C_9H_{19}$, and other products. Further, Lassieur (*Compt. rend.*, 1913, **156**, 795) reduced acetone above 200° over nickel; *iso*propyl alcohol and pinacone were not formed, but mesityl oxide and phorone were the intermediates, being subsequently reduced to methyl *iso*butyl ketone and di*iso*butyl ketone. Therefore nickel at higher temperatures gives abnormal products.

(d) *Chemical Methods of Reduction.*—In general, chemical methods of reduction of ketones give poor yields of alcohol or hydrocarbon, due to side reactions. For instance, with sodium or aluminium amalgam an aliphatic ketone is reduced to the corresponding secondary alcohol and a pinacone which may be the chief product.

Ziegler, Dersch, and Wollthan (*Annalen*, 1934, **511**, 13) prepared alkylbenzenes from alkyl aryl ketones by the Kishner–Wolf method of reduction. The semicarbazone is heated with sodium in absolute alcohol at 180° C. in an autoclave for 14 hours. They found much difficulty in the reduction of higher ketones. The yields of alkylbenzenes obtained, except amylbenzene, are not given.

Clemmensen's method (*Ber.*, 1913, **46**, 1838; 1914, **47**, 683) of reduction by means of zinc amalgam and hydrochloric acid is in general use for the preparation of hydrocarbons from ketones. Simon (*Bull. Soc. chim. Belg.*, 1929, **38**, 47), using Clemmensen's method, obtained amyl- and hexylbenzenes in 54 per cent. theoretical yield.

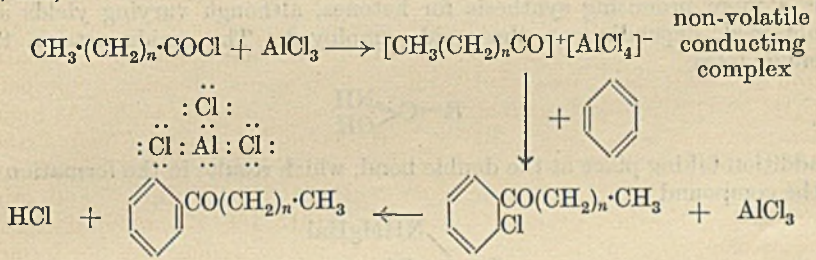
In view of the above, it may be concluded that palladium is the best catalyst and in chemical methods zinc amalgam is the best reducing agent. Both methods are being used in this laboratory for the reduction of alkyl aryl ketones.

Methods for the Preparation of Alkyl Aryl Ketones.

Alkyl aryl ketones of higher molecular weight are not listed. The methods employed for their preparation are numerous. Certain of these methods are compared below.

(a) *Acylation of Benzene by Friedel and Crafts' Reaction.*—Acylation by this process is a very reliable method with no rearrangement of alkyl group; this stability of the addend is readily understood on consideration of the mechanism given below. It is applicable to a variety of reactants (cf. Galloway, *Chem. Rev.*, 1935, **17**, 327).

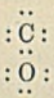
The co-ordinate compound of the acyl chloride and aluminium chloride initially interacts with the aromatic hydrocarbon at the double bond, which addition is immediately succeeded by the elimination of hydrochloric acid; further, the aluminium chloride forms a stable addition compound with the product of the reaction. In other words, the aluminium chloride labilizes the chlorine initially (cf. Schaarschmidt, *Z. angew. Chem.*, 1924, **37**, 286) :—

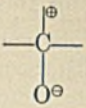


Simons, Randall, and Archer (*J. Amer. chem. Soc.*, 1939, **61**, 1795–1796) have demonstrated the effective use of hydrofluoric acid in the synthesis of ketones from aromatic compounds and carboxylic acids, anhydrides, and acyl halides. The yields increase with increasing amounts of hydrofluoric acid.

(b) *Method Employed by Sabatier.*—Sabatier and Mailhe (*Compt. rend.*, 1914, **158**, 830) prepared a series of alkyl aryl ketones by passing a mixture of the fatty and benzoic acids in the vapour phase over manganous oxide at 400–450° C. The method is only convenient for preparing ketones of lower molecular weight, on account of the higher fatty acids vaporizing with difficulty. The physical data recorded for *n*-octyl phenyl ketone prepared by Sabatier and Mailhe from pelargonic acid, however, are not in agreement with our own observations. The method is closely related to the classical method (Williamson) of the preparation of ketones by distillation of the calcium or barium salts of fatty acids under reduced pressure. Sabatier in his work " La Catalyse " mentions the catalytic formation of mixed ketones by the elimination of carbon dioxide from benzoic acid and lauric acid or myristic acid or stearic acid, or even phenyl acetic acid using thoria or manganous oxide as catalyst. The formation of three ketones and their separation present difficulty in obtaining a particular ketone in a pure condition.

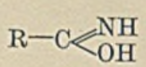
(c) *Grignard Synthesis of Ketones.*—The interaction of acyl anhydrides and alkyl magnesium halides to produce ketones has been the subject of extensive investigation. Ketones are produced in addition to tertiary alcohols. Again, by the carefully regulated action of magnesium complexes on acid chlorides and vice versa it is possible to prepare ketones, but here the reaction is not sufficiently controllable to give ketones in good yields (Nicholas and Wood, Investigation, Birmingham University). In explanation, it may be stated that the *negative* hydrocarbons ions, alkyl or aryl ($\overset{-}{\text{alkyl}} + \overset{+}{\text{MgHal}}$), attach themselves readily to the positively polarized atoms of carbon of the carboxyl group





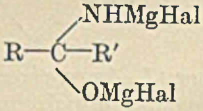
resulting in the easy formation of a tertiary alcohol. In both these cases it is difficult to stop at the intermediate ketone stage.

Possibly the action of acid amides on an excess of the Grignard reagent is a more promising synthesis for ketones, although varying yields are obtained, depending on the amide employed. The amide acts in the enolic form



$$\text{R—C}\begin{matrix}\text{NH}\\ \text{OH}\end{matrix}$$

addition taking place at the double bond, which results in the formation of the compound



$$\text{R—}\overset{\text{NHMgHal}}{\underset{\text{OMgHal}}{\text{C}}}\text{—R}'$$

which on treatment with acid or water readily decomposes to give the ketone.

This reaction should be investigated further; although there is a limitation to the chain length of R in formation of the Grignard reagent R–Mg–I and the use of the iodide is preferable. (As R increases, so the yield of the magnesium compound decreases, with simultaneous formation of olefine and saturated hydrocarbon.)

Yet the use of stearamide and palmitamide (cf. Ryan and Nolan, *Proc. Irish Acad.*, 1912, 30, 1) to form ketones opens up the possibility of the formation of *long* straight-chain or branched-chain ketones, and hence of straight-chain or branched-chain paraffins of definite constitution.

The use of nitriles of the type of benzonitrile C_6H_5CN is also possible, but with nitriles of the type of benzyl cyanide, $C_6H_5CH_2CN$ (which approximates in properties to an aliphatic nitrile), much poorer yields are obtained. Thus the length of the chain $C_6H_5(CH_2)_nCN$ attached to the benzene nucleus, both in this respect and also with respect to the methods of preparation of such semi-aryl cyanides, is strictly limited.

Other methods—for instance, the action of carbon dioxide under certain conditions on the Grignard reagent and the action of organo-magnesium halides on anilides and free acids—have been surveyed, but are not favourable for good yields of ketones of the type required in this investigation. It is difficult to arrest the reaction at the formation of ketone stage.

It is important to note that the *normal* action of a zinc organo-metallic compound on an acid chloride results in ketone formation with good yields :—

$$\mathrm{R{\cdot}COCl} + \mathrm{Zn}\Big<{}^{\mathrm{R}'}_{\mathrm{I}} \longrightarrow \mathrm{RCOR}' + \mathrm{Zn}\Big<{}^{\mathrm{Cl}}_{\mathrm{I}}.$$

The authors wish to thank Professor A. W. Nash for his continued interest in this work, which is being published in three papers. He has given every facility for the carrying out of the research and the authors are grateful to him.

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The University,
Birmingham.

OBITUARY.

VICTOR HENNY.

Dr. Victor Henny, European representative of Universal Oil Products Company, died at his home near London, on 12th July, 1940. He was born at Salatiga, Java, in 1887, and was educated in Switzerland. He attended the Technical University at Zurich from 1906 to 1910, obtaining a degree in chemical engineering. Three years later he gained the degree of Doctor of Science in Chemistry at the same university.

After leaving Zurich, Dr. Henny joined the staff of the N.V. de Bataafsche Petroleum Co. in Holland, and for two years was Acting Refinery Manager of the benzine installation of this company at Rotterdam. In 1915 his services were loaned by the Royal Dutch-Shell to England, and he worked under the late Sir William J. Pope, at Cambridge, on the production of toluene and mononitrotoluene from special Borneo benzine. Later he was Works Chemist and Works Manager at H.M. Factory at Sandycroft. In 1919 and 1920 he managed the Portishead Refinery of the Asiatic Petroleum Co., and was then sent to California, where he worked in the Shell Refinery at Martinez. Throughout this period he was making an intensive study of cracking processes in U.S.A.

In 1924 he returned to London and was placed in charge of the activities of the Universal Oil Products Company in Europe, Asia, Africa, and British Colonies. His early scientific education and refinery experience, together with his extensive knowledge of the languages and customs of various countries, aided by a rare charm of manner, made him exceptionally suitable for this appointment. Thus, he was able to develop rapidly the cracking of oils in Europe by the Dubbs process. From the first unit built at Cernavoda for the Omnium International de Pétrole in 1925, of 500 barrels, to the time of his death, some 100 units, of approximately 285,070 barrels daily capacity, with 41 licensees, were installed.

Dr. Henny took an active interest in the Institute of Petroleum, of which he was a Fellow. In addition, he was a Fellow of the Institute of Fuel, a Member of the American Chemical Society, a member of the American Chamber of Commerce and the Dutch Club. In recent months he was much concerned about the fate of the country of his birth, and he participated actively in the forming and direction of the work of the Netherlands Emergency Committee. Only three weeks before his untimely death he was presented to Queen Wilhelmina as a result of his work in this latter connection.

Dr. Henny leaves a widow and four children, to whom go the sincere condolences of all who knew him. The library of scientific books which Dr. Henny had accumulated during his career has been generously presented to the Institute of Petroleum by Mrs. Henny.

ABSTRACTS.

AUTHOR INDEX.

The numbers refer to the Abstract Number.

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

Geology and Development.

1320.* Wildcat Drilling in 1939. F. H. Lahee. *Bull. Amer. Ass. Petrol. Geol.*, 1940, **24**, 953–958.—Wildcat drilling was less intensive in 1939 than in 1938 and fewer pools were discovered.

From tables supplied in this review it is shown that during 1939 a total of 8,624,602 ft. was drilled in 2589 holes, making 270 producers and 2319 dry holes. Thus, 10·43% of the holes drilled and 12·87% of the footage were successful, the average depth of hole being 3331 ft.

G. S. S.

1321.* Developments in Eastern Interior Basin, 1939 and First Quarter of 1940. A. H. Bell. *Bull. Amer. Assoc. Petrol. Geol.*, 1940, **24**, 959–969.—The Eastern Interior Basin occupies an area of approximately 49,000 sq. miles, of which 37,400 sq. miles (76%) are in Illinois, the remainder being in south-west Indiana and in Western Kentucky. The greater part of the recent oil activity in the basin has also been in Southern Illinois.

The paper briefly discusses recent developments, special reference being made to the new Devonian limestone production in Western Illinois. Five pools were discovered : Sandoval, Salem, Bartelso, Centralia, and Tonti.

The top of the Devonian Limestone is from 1100 to 1400 ft. below the McClosky oolitic limestone, and probably exceeds 2000 ft. in the deepest part of the basin.

All the new Devonian pools are in areas of structural closure as shown on either Pennsylvanian or Mississippian key beds or both ; moreover, they are in areas of previous production from shallower sands. Initial production of these wells is exceptionally high for the area, exceeding in some cases 10,000 brl. per day.

G. S. S.

1322.* Deep-Sand Developments in Appalachian Region during 1939. T. H. Myers. *Bull. Amer. Assoc. Petrol. Geol.*, 1940, **24**, 970–973.—Developments for deep-sand oil and gas in the Appalachian region during 1939 had disappointing results. In Southern New York and Northern Pennsylvania each provided two new Oriskany gas-pools, but they all proved of very limited extent. Similarly, in Jackson Co. (West Virginia) two Oriskany pools were discovered, but neither has so far been tested sufficiently to give indication of its extent.

A map of the Appalachian area showing Oriskany development accompanies the paper.

G. S. S.

1323.* Developments in Michigan during 1939. R. B. Newcombe. *Bull. Amer. Assoc. Petrol. Geol.*, 1940, **24**, 974–993.—During 1939 the leading fields in Michigan were : Bloomingdale (Van Buren Co.), Temple (Clare Co.), and Walker or Grand Rapids (Kent Co.).

The Bloomingdale field is the farthest south-west and the shallowest in depth of any size in South-western Michigan. It was the most actively drilled field in the State during the latter part of 1938 and the first half of 1939. Production is somewhat scattered and there are two non-productive saddles on the fold : Eastern Bloomingdale Township and along the Bloomingdale–Columbia Township line. Statistics of production are therefore given in three divisions : Bloomingdale, Columbia, and Mill Lake.

Temple field was discovered at about the same time as Bloomingdale, the major part of the producing area being in Redding. It is a " basin " field, production coming from a porous dolomitic zone just below the Bell Shale and considered to be of Monroe age.

The Grand Rapids field, although slow in developing early in the year, took on major proportions rapidly, and by June a boom was in progress. Production is from the Traverse at 1750–1900 ft. The limestone section in which porosity occurs is 30–50 ft. thick, but the actual pay section is much less. Large quantities of shallow gas have been found in the Berea (?) producing horizon at 1070–1100 ft., but no commercial use is made of the gas except for fuel in drilling. The Grand Rapids field has not been prospected beneath the Traverse on the top of the structure.

Other active fields in Michigan were Salem (Allegan Co.), Wise (Isabella Co.), Dundee or Deerfield (Monroe Co.), and the Wisner or Akron field in Tuscola Co.

Despite the discoveries, and increased activity in 1939, these did not keep pace with decline, and unless deeper production or new flush fields are found, it is considered that the outlook for 1940 is not particularly bright. G. S. S.

1324.* Developments in North Mid-Continent in 1939. E. A. Koester and R. F. Moyer. *Bull. Amer. Assoc. Petrol. Geol.*, 1940, **24**, 994–1010.—Drilling activity in Kansas decreased by about 14% compared with the previous year and by 48% below that for 1937. Initial oil production per well, however, increased from an average of 1208 brl. to 1577 brl. Norton and Phillips are now oil-producing counties, but in each area the discovery well is small and of doubtful economic importance. In Sherman Co. a gas discovery was made from the Cretaceous; this is of interest and importance, as this county was previously unproductive.

In Missouri forty-one wells were drilled in 1939, but, of these, eighteen did not reach Mississippian rocks. Twenty-three wells were drilled to horizons sufficiently deep to be considered " deep tests." Stratigraphically they include Pre-Cambrian (one), Arbuckle limestone (sixteen), Upper and Middle Ordovician (three), Devonian (two), and Mississippian (one). Results of drilling were definitely discouraging, but failure to produce, due to structural irregularities, should not be regarded as a true measure of the oil and gas possibilities of pre-Pennsylvanian rocks.

In Nebraska six important dry holes were completed, none of which is in the Forest City basin. The discovery of oil in the Pawnee Royalty Co.'s Boice No. 1, in Sec. 18, T. 1 N, R. 16 E, was probably the most encouraging development in Nebraska, although at present the well has not been successfully completed as a commercial producer.

Some drilling activity took place in the basin part of Iowa, but no completions were made. Wells in Charles Mix and Union Counties (South Dakota) were completed in Pre-Cambrian rocks, and supplied much valuable stratigraphical data.

G. S. S.

1325.* Developments in Oklahoma during 1939. E. F. Shea. *Bull. Amer. Assoc. Petrol. Geol.*, 1940, **24**, 1011–1024.—Oklahoma in 1939 saw a slight increase in drilling operations, particularly in exploratory wells. New pools and extensions were thus added during the year, and showed an increase over the number recorded for 1938.

Little change occurred in the amount of geological and geophysical work carried out in the State, although exploratory operations were conducted in outlying areas away from producing pools.

Total production for the State during 1939 was 153,500,000 brl., a decrease of 16,500,000 brl., or 9·7%, on the previous year's total. Other than in the St. Louis–Maud area all the major producing pools were concerned in the general decline.

Of a total of 2065 completions, 268 (13%) are classified as exploratory wells. Sixty new discoveries were made; thirty-one pools and twenty-nine extensions to old pools. These are distributed over twenty-four counties: Seminole, with eleven areas, and Pottawatomie, with eight, predominating.

Stratigraphically they are distributed as follows: thirty-five in Pennsylvanian deposits, eleven in Siluro–Devonian, and fourteen in Ordovician. Despite the many discoveries of 1939, only three appear to be particularly noteworthy, viz. Hobart Pool (Kiowa Co.), Byars Pool (McClain Co.), and the Arbuckle production in the Frederick pool (Tillman Co.). G. S. S.

1326.* Will Illinois See Drastic Drop by Year's End? Anon. *Oil Wkly*, 22.7.40, **98** (7), 10.—Illinois' production is expected to decline, due to a fall in the Devonian output of Salem and to a fall in the Benoist, McClosky, Stein, and Aux Vases production throughout the State. The production history of the Devonian at Salem and Centralia is given in tabular form.

In general Devonian wells stop flowing in 1 to 3 months. At Centralia 72% of the Devonian wells are already on the pump.

At present there seems little hope of large discoveries. A 200-brl. well has been completed in the Trenton at Salem, and many Devonian wells could readily be deepened to this horizon. There is a possibility of Devonian development at Loudon.

G. D. H.

1327.* Drilling Comparatively Brisk Despite Curtailment. L. J. Logan. *Oil Wkly*, 5.8.40, **98** (9), 10.—The July drilling was less intense than that of June, but greater than that of the corresponding period of last year. So far this year completions have been nearly at the 1937 level.

Tables give the total completions for the first seven months of the years 1933–1940, and the number of wells drilling in various States at the beginning of August as compared with the same date in 1939. The numbers of completions and their status are given in other tables.

G. D. H.

1328.* North-eastern Ohio Attracts Major Interest. S. W. Tait. *Oil Wkly*, 5.8.40, **98** (9), 32.—North-east and south-east from Cleveland some 200,000 acres of land have been leased, and two tests have been started in this region. Geophysical crews are at work.

The present gas-fields of Ohio are so depleted that they supply only a fraction of the domestic demand. There is a good knowledge of the geology as a result of the wells drilled during the past 80 years. The regional dip is south-east into the Appalachian geosyncline. Typical wells penetrate Mississippian, Devonian, and Silurian beds, which have oil and gas horizons. The possible producing horizons other than the shallow Big Injun are the Berea, Newbury, and Clinton. The Oriskany offers possibilities, but its occurrence is erratic. Ordovician tests in the Eastern fields offer little encouragement.

G. D. H.

1329.* Careful Exploration Required in North Dakota Oil Search. F. C. Foley. *Oil Gas J.*, 18.7.40, **39** (10), 14.—So far the search for oil and gas in North Dakota has yielded gas alone, and that in only one area.

Pre-Cambrian rocks do not outcrop in North Dakota, but are only a few hundred feet deep in the east. Palæozoic beds underlie all but the south-east corner, where Cretaceous rests directly on Pre-Cambrian. The Palæozoic thickens westwards to a maximum of about 6000 ft. Pre-Cretaceous Mesozoic is known which also thickens westwards. Lower Cretaceous beds are missing, but the Dakota occurs, followed by the Benton, Niobrara, and Pierre shales, calcareous beds and lenticular sandstones. Most of western North Dakota is underlain by Fort Union sands, clays, and lignites. Small patches of Oligocene are known.

The main structural feature is the Williston basin, a continuation of the Lemmon geosyncline of South Dakota. It may have a thickness of 12,000–14,000 ft. of sediments. Subsidiary structures may occur on the flanks of the Williston basin, but the surface evidence is not very satisfactory. The Cedar Creek anticline runs into south-western North Dakota, and has given gas. Geophysical work has been carried out in search of suitable structures for oil accumulation.

It is believed that oil in quantity may be confined to pre-Cretaceous beds, although the Cretaceous has shown oil and gas.

G. D. H.

1330.* Production Exceeds New Proven Reserves So Far This Year. W. V. Howard. *Oil Gas J.*, 25.7.40, **39** (11), 55.—During the first half of 1940 the U.S. proven reserves declined by 409,045,094 brl. Only Kansas and Mississippi found new reserves in excess of their production. It seems probable that many groups of small pools will be joined into single large pools, with a consequent increase in estimated reserves without much additional drilling.

A table shows the production, new discoveries, and extensions to old fields in terms of barrels. A second table gives the proven acreage, formation thickness, and estimated reserves for the new discoveries and extensions in the different States.

G. D. H.

1331.* World Crude Oil Production 6,000,000 Barrels Daily. H. S. Norman. *Oil Gas J.*, 25.7.40, **39** (11), 56.—During the first half of this year world crude-oil production attained a new peak. The main increases were in countries still at peace. World production rose by 7·6%, of which the U.S.A. contributed 6%.

Saudi Arabia's production rose by 119% to 1,180,000 brl., and Hungary's rose by 114%. Iraq's production has fallen by 50% since the middle of June.

The production in the first six months of 1939 and 1940 is tabulated for the different countries, and a map shows the locations of the main European and East Asiatic oil refineries, the capacities and cracking capacities of which are presented as totals for the various countries. G. D. H.

1332.* Rincon Field of South Texas Being Steadily Extended. N. Williams. *Oil Gas J.*, 1.8.40, **39** (12), 13.—The producing area covers 1800 acres, and there are fourteen oil sands. This is probably only a quarter of the potentially productive area. Thirty-six oil and four gas-distillate wells have been drilled. Oil is obtained from nine sands at 3500–4300 ft., and gas-distillate from 4800 ft.

The structure appears to be a dome elongated north-east–south-west. It is 4–4½ ml. long and 3–3½ ml. wide. At the crest are 600–800 acres of gas and distillate production, whilst down dip oil is developed. The present development is mainly on the north-west and north, but some wells have been completed on the south-west of the structure. Each of the fourteen sands was carefully tested in a single well. All the sands are not found in every well, but most wells have more than one sand. The sands range in thickness from 10 to 60 ft. The first nine sands are in the Frio. Where several sands occur in one well they are produced successively from the bottom upwards.

The proved reserves are approximately 40,000,000 brl., and the wells are rated at 45–50 brl./day, according to depth. The average bottom-hole pressure is 1700 lb./in.^{2}. Two of the wells are on gas-lift; the rest flow.

The history of development is briefly outlined. G. D. H.

1333.* Mississippi Entering Settled Exploration Stage. G. Weber. *Oil Gas J.*, 8.8.40, **39** (13), 22.—Since April there has been a drop in Mississippi drilling, and there has been a low degree of success in recent exploration. Many of the wildcats are deemed to have been poorly located and too shallow. Leasing and geophysical work have also declined.

Blanket sands occur in the Selma Chalk, Eutaw, and Tuscaloosa throughout a wide area. There are few outstanding structures like Jackson or Tinsley, but less pronounced structures have been shown geophysically—*e.g.*, the productive Pickens structure.

There has been much interest in the North-west Alabama basin, with its producing possibilities in the Palæozoic. Asphalt occurs in outcropping Mississippian beds. Geophysical work has been going on, and some wildcats are under way.

Southern Alabama has been sparsely drilled. It has gas shows in the upper beds. Two deep tests are to be extended through the Selma.

Tinsley has a recoverable reserve estimated at 24,000,000 brl. The main production is from the Woodruff sand at the base of the Selma Chalk. The sand thickens to the north-west, north, and east. Whilst the potential is 37,500 brl./day, the production averages 8000 brl./day. A major fault runs north-east–south-west through the field. There are productive possibilities in the underlying Eutaw, and this yields oil in the south-west where the Woodruff is missing. A sand between the Woodruff and the Stevens of the Eutaw also yields oil. Faulting makes subsurface correlation difficult.

Pickens has four producers, and it is thought that the present production is from the north-west flank of the structure. G. D. H.

Drilling.

1334.* Drilling in United States. Anon. *Oil Gas J.*, 25.7.40, **39** (11), 76.—A statistical study of drilling in the various States of America is made. It is shown that an increase of 2586 in the number of wells completed in the U.S.A. during the first half of 1940 as compared with the same period of 1939 brought the total number of completions to 15,053—only sixty-nine below the number recorded for the record year of 1937. A. H. N.

1335.* Rock-bit Efficiency and Study of Tool-Joint Life. V. V. Horner. *Oil Gas J.*, 25.7.40, **39** (11), 171.—*Paper presented before American Petroleum Institute.*—After a detailed analysis of performance in the Louden field, it is concluded that drilling costs may be materially reduced if the following tactics are adopted in analysing the facts in hand.

For bit efficiency: (1) Detailed records should be maintained for individual wells regarding bit performance, such as footage/bit, hours on bottom/bit, and well average footages/bit and penetration. (2) To determine the proper type of bit for efficient operation, types of bits should be compared as used in numerical order. (3) The economic range of bit footage and penetration rate should be ascertained by establishing the relationship between the two and its effects on total cost, and with the choice of wells the averages of which fall within this range, optimum footage and rate for each separate bit in the sequence should be determined.

For tool-joints studies, periodic inspection with orderly compilation of data pertaining to outside-diameter wear and condition of tool-joint faces and threads can be utilized in providing comparative means of controlling tool-joint maintenance costs.

That this procedure is applicable in an area where stratigraphic characteristics are more or less regular and where activity is stimulated to the extent of providing initial data has been indicated by the results at Louden. A. H. N.

1336.* Application of Tapered Roller Bearings to Oil-field Uses. S. M. Weckstein. *Oil Gas J.*, 1.8.40, **39** (12), 38.—The importance of selecting the proper bearings for oil-field equipment where conditions of working are particularly strenuous is emphasized. The principles and mechanics involved in the design of a tapered roller bearing are briefly outlined and illustrated.

Typical tapered roller-bearing constructions are discussed, together with their respective advantages. These bearings are adaptable to carry all kinds of loads, radial, thrust, and all combinations of the two simultaneously. It is claimed that shafts run true and are kept permanently aligned.

The use of tapered roller bearings in reverse clutches, draw-works, and rotary tables is studied. In all cases, by replacing the sliding friction of plain bearings with smooth rolling friction less power is consumed in driving the machinery; dependable, trouble-free service is more nearly assured; and faster drilling is made possible, with lower maintenance and repair costs. A. H. N.

1337.* Rotary-Rig Foundations in Gulf Coast Marshes. N. Williams. *Oil Gas J.*, 8.8.40, **39** (13), 42–43.—Design and construction of foundations for rotary rigs operating on marshy locations are being given more study by Gulf Coast companies as depth of drilling increases, with consequent use of heavier equipment. Piling and mats are widely used, but are usually subject to uneven settling under excessive loads, or weakening by vibration. At a recently designed job in one Gulf Coast field the principles of construction employed gave greater strength, support, and rigidity to the piling foundations, thereby reducing the piling and bracing required. Thus costs of building were substantially reduced.

A plan and elevation of the newly designed piling foundation are included, as well as a discussion of the various aspects of design considerations to be taken in such an undertaking. A. H. N.

1338.* Combating Loss of Circulation in Drilling and Cementing. J. B. Stone. *Oil Gas J.*, 8.8.40, **39** (13), 44–45.—A new method of preventing fluid loss both in drilling mud and cementing slurry circulation consists of using a flaky organic material, mixed in relatively small proportions in the cement slurry or mud. Its use is claimed to be particularly successful.

The material is a chemically inert product, fabricated into small, paper-thin flakes. It is not materially deteriorated or softened by water, oil, mud, or cement slurry. The plates are tough and strong even when wet. The flakes are specially treated to give them a crinkled surface, so that they will not adhere together when wet. The density of the material is similar to that of mud, so that they will not settle.

The dimensions are given as 0·001 in. thick, and in an irregular shape from ½ to 1 in. in average length. The material passes easily through the pumps. As the flakes are flexible and pliant, however, they form a sheath or cover over the rough and irregular surface of a formation by being strained out of the circulating fluid.

Results of tests made on various materials are given. A. H. N.

1339.* Drilling Time Lost by Crooked Holes. T. P. Sanders. *Oil Gas J.*, 8.8.40, **39** (13), 55.—It is pointed out that in measuring deviations in a bore-hole the tests are usually made too far off the bottom. Thus in a case cited, when 2½° were measured, actual deviation of the well at bottom was 3¾°. To avoid this difficulty it is recommended that : (1) If a float valve is used in the drill stem, it should be placed immediately above the drill collar, so that the instrument can be run as close to the bit as possible. (2) The deviation tests should be made immediately after adding a new joint, so that the bit will be practically on bottom at the time of test. (3) Tests should be made frequently, probably every 50–200 ft., depending on conditions. (4) The accuracy of the instrument and methods used should be sufficiently high to show deviations of 0·5° with certainty. A. H. N.

1340.* Squeeze Cementing Application and Technique. P. D. Torrey. *Oil Wkly*, 29.7.40, **98** (8), 68.—The conclusions reached from this fairly long paper are : Squeeze cementing in lime formations has recently shown good results, in great contrast to previous results. Excellent results have been obtained in West Texas fields in shutting off gas by the use of chemical plugging agents.

Smaller quantities of cement, when properly controlled, appear to be giving even better results than the larger batches formerly used.

The average percentage of success in squeeze cementing has definitely improved, and is now in the neighbourhood of 70% for all jobs. Some squeeze jobs have been attempted for the exclusion of water in essentially depleted reservoirs. The chances of success for such conditions are, however, remote.

The tendency for squeeze cementing before completion for reduction of gas/oil ratios is increasing, and is apparently giving better results than where squeezing is delayed.

It is believed that the speed with which large quantities of heavy cement slurry drop into the tubing with the circulation joint open causes a fluid-hammer effect which has been responsible for the collapse of casing. The substitution of smaller quantities of cement used in multiple-batch cementing may assist in correcting this difficulty.

Water exclusion in the East Texas field continues to be a problem. A relatively small number of wells still contribute the greater percentage of the water produced, and in view of the probable high degree of depletion of those areas more seriously affected by water, it is doubtful whether corrective measures under such conditions will prove to be profitable. A. H. N.

1341.* Drilling Activities. L. J. Logan. *Oil Wkly*, 5.8.40, **98** (9), 10–12.—Statistical data for drilling during July 1940 are presented and contrasted with corresponding periods of previous years. The paper deals with activity in the various States of the U.S.A. only. A. H. N.

1342.* Exact Measurement of Casing While Running into Hole. H. E. Gross and J. P. Hays. *Oil Wkly*, 5.8.40, **98** (9), 20.—In cases where casing measurement can be checked by independent methods—such as where the casing is landed on bottom of hole—the two independent figures for the depth should check to within 1 ft. in 4000 ft. It is reported that in a deep well where careful measurements were essential duplicate measurements checked to 0·11 ft. in 10,700 ft., whilst 1 ft./10,000 ft. is not uncommon.

Methods for precise measurements of casing both in the racks and when being run into the well are described in exhaustive detail. The necessity of attention to every step in the procedure is emphasized. Troublesome items and pitfalls are stressed.

A procedure is also given for "juggling" pipe—*i.e.*, from the pipe at the top of a well, joints may be so selected or rejected as to total a predetermined length. An example is worked out to illustrate this procedure.

It is pointed out that a paper on depth measurement by means of multiple-strand wire lines appeared in *Oil Weekly*, 22.7.40; and one on drill-pipe and tubing measurements in the same *Journal* of 13th, 20th, and 27th June, 1938. These five papers describe all essential measurements involved in oil wells. A. H. N.

1343.* Oil-field Boilers. Anon. *Oil Wkly*, 19.8.40, **98** (11), 19–26.—The paper deals with trends in oil-field boiler design and use, and is illustrated by photographs of, and explanatory notes on, the main types of boilers commonly found in practice.

During the past few years boiler manufacturers have stepped up the rated working pressure of their top-line steam-generating units to a generally accepted high rating of 350 lb./sq. in., meanwhile holding the steam-generating unit within the limits of oil-field portability and overall dimensions. More recently working pressures of 500 lb./sq. in. have been utilized.

The steam dome, borrowed from locomotive-boiler practice where changes in speed and direction produced piling-up of the water, due to inertia forces, against one or the other tube sheets, seems to be disappearing from the oil-field unit.

Steam-liberating area is being increased. Foaming and other undesirable steaming characteristics increase in severity as steam-liberating area is restricted.

The controversy over superheat is apparently not yet settled. The two aspects of the question are discussed; both types of units are available—*i.e.*, with and without a superheater.

Various aspects of boiler design are also reviewed. A. H. N.

1344.* Light-Weight Drilling Equipment. H. L. Flood. *Petrol. Engr*, August 1940, **11** (12), 27–28.—Drilling practice in Illinois is specially studied. More important than any other factor as an influence on the manner in which drilling is conducted in Illinois and on the type of equipment used is the matter of shallow depths. Thus far 2000–3000 ft. are the average depths to which most of the wells have been drilled.

The drilling equipment in use in these areas is predominately of the light-weight, easily moved type generally classified as slim-hole or streamlined equipment. Many can be winched on a truck and readily moved as one piece. Equally important are the truck-mounted portable masts and drilling machinery generally classed as being of the core-drilling type.

Several entirely new designs in spudders recently introduced by various manufacturers are now being extensively used in the Illinois fields.

The general practice is to drill down with rotary equipment to the top of the producing formation, set and cement casing, move out the rotary equipment, and then move in a spudder to drill out the plug and complete the well. A. H. N.

1345.* High-Pressure Salt-water Flow in Drilling Operations. M. L. Cashion. *Petrol. Engr*, August 1940, **11** (12), 90.—When a drilling well encounters high-pressure salt-water flows it usually means the abandonment of the operation if only orthodox methods are employed in an endeavour to combat such flows. In an area on the Gulf Coast twenty tests have had to be abandoned because of inability to cope with the salt-water flow always encountered under excessive pressure at depths of about 10,000 ft. The paper describes the procedure of combating this danger employed in a well recently successfully drilled to 12,000 ft. in the same area.

The success of the operations was probably due to the fact that preparations were made for every possible contingency, and it is now decided that in future operations none of the apparently too numerous precautions taken will be eliminated.

The two chief items of interest, however, were the pressure-drilling technique used and the mud treatment. Relatively few innovations were employed. The pressure-drilling equipment and technique were directed towards constantly maintaining a back-pressure on the salt formation. The mud treatment prevented excessive flocculation of the colloid under the influence of the salt water.

Details of both these items are fully given. The pressure equipment included: (1) hydraulic feed rotary, including snubbing equipment; (2) pressure-control equipment on the well-head; (3) chokes on the mud return lines; (4) back-pressure valves, etc., in the drilling string; and (5) external-flush drill pipe, to permit going in and out of the hole through the drilling packer under pressure. Each item is discussed separately.

In the mud used, silicate of soda was included as an ingredient to counteract the effects of high-pressure salt-water flows. Furthermore, the liquid phase weighed 11·8 lb./gal., against 8·33 lb./gal. in the case of ordinary water muds. The extra weight in the liquid phase allows fabrication of high-weight muds with a much lower suspended–solids content than is ordinarily needed. Various other advantages of this silicate mud are discussed.

The paper deals further with the technique and other incidental equipment used, which nevertheless is believed to have been essential to the success of the project.

A. H. N.

1346. Drilling Patents. C. D. Barnes. U.S.P. 2,209,591, 30.7.40. Appl. 20.1.36. Well-drilling fluid containing starch and an aliphatic alcohol, besides the heavy solids suspension characterized by low penetration and low gas-cutting properties.

C. E. Wilcox. U.S.P. 2,209,619, 30.7.40. Appl. 3.9.37. Cable-tool drill-bit having a pilot cutter of smaller diameter than the intended hole and a reamer eccentrically disposed to the vertical axis.

C. C. Matlock. U.S.P. 2,209,988, 6.8.40. Appl. 4.3.39. Pipe tongs.

B. Hanly. U.S.P. 2,210,077, 6.8.40. Appl. 12.10.39. Drill-bit cone-cutter bearing arrangement.

T. H. Stancliff. U.S.P. 2,210,101, 6.8.40. Appl. 30.1.39. Drilling bit for rotary rigs.

O. Duda. U.S.P. 2,210,202, 6.8.40. Appl. 26.2.38. Travelling block construction.

A. C. Catland. U.S.P. 2,210,279, 6.8.40. Appl. 18.2.38. Rock bit with cutters on horizontal ball bearings.

C. L. Henry. U.S.P. 2,210,407, 6.8.40. Appl. 2.9.38. Portable derrick, being hinged at one end so that it may be lowered horizontally for transport, and the upper sections adapted to be telescoped to shorten overall length.

C. L. Henry. U.S.P. 2,210,408, 6.8.40. Appl. 8.9.38. Marine-working platform structure initially supported on a floating barge, but arranged to be finally supported on firm bottom by means of towers and removing barge away from structure.

C. M. O'Leary. U.S.P. 2,210,464, 6.8.40. Appl. 20.9.37. Means for taking side-wall cores having in combination a surveying instrument for recording direction and inclination of hole simultaneously with coring.

R. T. Cloud. U.S.P. 2,210,795, 6.8.40. Appl. 16.2.39. Method and apparatus for electrically logging a bore-hole.

S. A. Linney. U.S.P. 2,210,815, 6.8.40. Appl. 23.1.39. Tool-joint pin.

B. F. Walker. U.S.P. 2,210,824, 6.8.40. Appl. 27.12.38. Rotary drilling tool for scraping the walls.

J. H. Williams. U.S.P. 2,210,826, 6.8.40. Appl. 19.4.39. Fluid packing for a swivel.

H. Deckard. U.S.P. 2,210,902, 13.8.40. Appl. 28.6.38. Fishing tool.

G. Kroll. U.S.P. 2,211,016, 13.8.40. Appl. 14.9.38. Elevator for supporting pipe, possessing a special hinge mechanism having horizontal and vertical hinging axes.

J. J. Jakosky. U.S.P. 2,211,124, 13.8.40. Appl. 11.3.39. Method for continuously exploring bore-holes while drilling by using the drilling string as an electrode, and measuring resistance of earth to a D.C. at various distances from bore-hole for each depth reached, and repeating operations at various depths.

E. J. Shaffer. U.S.P. 2,211,173, 13.8.40. Appl. 6.6.38. Pipe coupling, being a tool-joint arranged so that the box exerts a compressive stress on the pin; a welded ring made to avoid localized stresses.

A. L. Stone. U.S.P. 2,211,178, 13.8.40. Appl. 21.8.37. Method of making a joint-box on end of pipe.

A. L. Stone. U.S.P. 2,211,179, 13.8.40. Appl. 12.11.38. Casing joint for casings of equal inner diameter, one end of pipe being belled to receive the spigot end of the other.

J. A. Tytus and A. L. Stone. U.S.P. 2,211,183, 13.8.40. Appl. 12.9.38. Reamer for well pipe-joints.

M. B. Chittick and A. F. Schlandt. U.S.P. 2,211,254, 13.8.40. Appl. 3.2.37. Wire rope lubricant consisting of viscous mineral oil, fibrous material, and from 3 to 20% hydrocarbon polymers which oxidize to tough dry coatings on contact with air.

W. G. L. Smith. U.S.P. 2,211,299, 13.8.40. Appl. 4.1.37. Wire-cable guide.

A. N. Troshkin. U.S.P. 2,211,466, 13.8.40. Appl. 28.7.39. Hose coupling.

R. E. Lilleborg. U.S.P. 2,211,507, 13.8.40. Appl. 23.11.38. Self-adjusting pipe wrench.

H. T. Byck, J. W. Freeland, and H. C. Lawton. U.S.P. 2,211,688, 13.8.40. Appl. 12.7.39. Method of treating oil and gas wells, during drilling, with a fluid of water-soluble alginate, an alkali hydroxide, and a water-soluble organic material of weakly acidic properties and an alkali pyro- and meta-phosphate.

C. R. Irons. U.S.P. 2,211,696, 13.8.40. Appl. 23.9.37. Treatment of wells for the removal of mud-sheaths electrically.

W. A. Warburton. U.S.P. 2,211,803, 20.8.40. Appl. 7.8.39. Method and equipment for multiple whipstock drilling and lining.

C. Gauthier. U.S.P. 2,211,973, 20.8.40. Appl. 22.9.39. Fishing tool of the pin-tap type.

M. H. Joseph. U.S.P. 2,211,978, 20.8.40. Appl. 5.12.38. Hose coupling.

B. E. Parrish. U.S.P. 2,212,040, 20.8.40. Appl. 28.5.38. Oil-saver with a bore the size of which is reducible.

J. E. Hoffoss. U.S.P. 2,212,067, 20.8.40. Appl. 20.3.39. Drilling apparatus consisting of a drill-bit and a guide, the guide having a reaming tool incorporated in it.

C. W. Thornhill. U.S.P. 2,212,086, 20.8.40. Appl. 27.10.36. Float-collar and guide-shoe.

C. W. Thornhill. U.S.P. 2,212,087, 20.8.40. Appl. 27.11.39. Well-cementing apparatus utilizing the guide-shoe of U.S.P. 2,212,086.

V. B. Zacher. U.S.P. 2,212,108, 20.8.40. Appl. 23.4.38. Process for treating mud consisting of clay, water, and lignin. A. H. N.

Production.

1347.* Distillate Fields Bring New Production Phase. B. Mills. *Oil Wkly*, 29.7.40, **98** (8), 53.—A review is made of the theory and practice of distillate production involving retrograde vaporization and gasoline extraction and recycling of dry gas.

Retrograde condensation is the change of gas to a liquid state following a reduction of pressure. Pressure maintenance in a reservoir of gases in the retrograde pressure regions becomes necessary. A substantial reduction in formation pressure is usually accompanied by a sharp loss of fluid that could otherwise have been recovered as gas under the original reservoir pressure. Conversely, it may be possible, by increasing the formation pressure, to force into the vapour phase certain heavier products that were originally in the reservoir in the liquid state. A case is cited where this process has probably occurred.

The nature of diffusion of the injected gas with the wet gas already in place is not fully known, but it is believed that some mixing does occur. A few practical field-men believe that dilution or mixing is extensive, but it is known that proper spacing of injection wells will force the wet gas to the producing wells with a minimum of dilution.

Various plants are described. A. H. N.

1348.* Gas Lift Becomes Major Producing Method. Anon. *Oil Wkly*, 29.7.40, **98** (8), 62.—The trend of gas-lift installations appears to be towards wider application of differential type units in holes where thorough study indicates a wide variation in anticipated flow rates of the well, and where wide range of water content may be expected.

Intermittent lifts with either clock, pressure, or combined surface controls find a place in the same field as automatic intermitter, and fit particularly well into a lease wherein close regulation of the flow within stated limits must be maintained. For this type of production, with increasing water content, exchange of the master timing unit enables the intermitting frequency to be varied as conditions change.

Where ample gas volumes are available, either the plunger lift or the pneumatic flow-chamber may be used, although in both cases the time interval required between cycles may intervene to prevent either from handling the requisite volume of fluid from a well making a high percentage of water. A. H. N.

1349.* Deep Well Pumping. Anon. *Oil Wkly*, 29.7.40, **98** (8), 89.—This paper represents the views of many contributors on two questions put to them by the *Oil Weekly* regarding the best method of artificially lifting oil from 10,000-ft. or deeper wells. A summary of the answers is as follows : (1) A general inclination to leave the solution of the problem until it arises. (2) Fairly general agreement that all available means should be utilized to extend the proportionate quantity recovered by natural flow, and thus at least delay the necessity of artificially lifting oil from such depths. (3) Because most deep wells may be expected to maintain a high fluid level in the well after natural flow ceases, it will rarely be necessary to lift oil from depths actually exceeding present lowest pumping levels. Therefore current pumping methods may be used. (4) Should it become necessary to lift from the bottom of 10,000—13,000-ft. hole, the majority of engineers at present think in general terms of some bottom-hole pump actuated by liquid pressure applied from the surface, by means of gas lift, standard pumping, electric centrifugal bottom-hole pumps or a combination of gas-lift with bottom-hole units. A. H. N.

1350.* Modern Equipment Keeps Lifting Costs Low. E. Sterrett. *Oil Wkly*, 29.7.40, **98** (8), 96.—Typical of the trend towards smaller and cheaper pumping units is the entry of the pumping-unit type of machine into the light or stripper well fields of 5-h.p. loads, the adoption of metal-to-metal pumps in the medium- and low-price fields, and the use of hydraulic pumps in small-capacity wells. Other improvements in well-servicing equipment built for safe and rapid work are discussed.

Large units are further described and their advantages enumerated. Details are given of the units mentioned in the first paragraph. Improvements discussed in the case of centrifugal submerged pumps are the glass insulation for motor windings, flat cable conductors, and the use of corrosion-resistant parts, all adapted with a view to lengthening the normal life of the unit.

Cushions for rod-strings are of three types: (1) those incorporating resilient solids such as springs or rubber; (2) those using compressibility of air or gas; and (3) those using controlled fluid flow under pressure to regulate the relative motion between linked parts.

Methods of combating rod corrosion and sand form the conclusion. A. H. N.

1351.* Technical Advancements and Economics of Water-Floods. L. M. Arnold and R. C. Earlougher. *Oil Wkly*, 29.7.40, **98** (8), 105.—The basic fundamentals of water-flooding having been established, the problem now is how to use the method economically. It is found to be cheaper in the long run to use siliceous lime-water than river-water. This is due to the variation both in quantity and quality of river-water, necessitating continuous watching and checking. Once a process of treating siliceous lime-water is set up it may be considered to be stable and relatively automatic. The chief drawback in using this water is its relatively high corrosiveness. This difficulty is being overcome by using cement-lined pipes.

The water-to-oil ratio averages 10 : 1, being between 5 : 1 and 18 : 1 in various projects.

Another operating method which is being used in Mid-Continent area is to flow the oil from the wells rather than pump it. This is accomplished by using higher water pressures, and results in low initial investment, due to absence of pumping units. Discussions are still in progress as to which is the better method: flowing or pumping.

A strong plea is made for wide well spacing from an economical point of view, and a short discussion is given on the economics involved in spacing programmes.

A. H. N.

1352.* Distillate Fields Bring New Production Phase. Part II. B. Mills. *Oil Wkly*, 5.8.40, **98** (9), 13–19.—Recycling methods involve the adaptation of methods falling into one of the following classifications: separation, refrigeration-separation, and absorption. Descriptions are given of recycling plants employing the straight separation method. The principle of all such plants is to run the gas into one or more separators in series, reducing the pressure and recovering the liquid distillate.

Flowsheets, photographs, and pertinent data accompany the descriptions of plants used in Stratton field, Long Lake, and Bammel field, all in Texas.

In the last-mentioned plant gas from wells is run through chokes and meters to two high-pressure separators, the lean gas from which is scrubbed in a tower, compressed, and reinjected into an input well. The bottoms from the high-pressure separators are passed through intermediate and low-pressure separators and finally stored, the lean gas from these last separators being used as fuel. A. H. N.

1353.* Mobile Pump House for Flood Project. F. B. Taylor. *Oil Wkly*, 12.8.40, **98** (10), 17–18.—In a flooding project using river-water it was found that the river could obtain a stage variation of 40 ft. To obviate possible damages from shutdowns, the problem of protecting the water-supply system at all times was solved by mounting the pump-house on an inclined track and rendering the pump mobile.

A description is given of the construction and operation of the unit. A. H. N.

1354.* Distillate Fields Bring New Production Phase. Part III. B. Mills. *Oil Wkly*, 12.8.40, **98** (10), 19–28.—In this part of the series of articles on recycling, descriptions are given of plants using temperature reduction for extracting liquids from distillate production, and commonly called refrigeration–separation systems.

Flow-sheets, photographs, and pertinent data accompany the descriptions of the several plants. The process in one of these may be summarized: Gas passes through a cooler and into a scrubber, from the top of which it goes into a dehydrator, where it meets a calcium–chloride solution cooled by an ammonia refrigeration plant, after which it passes on to high- and low-pressure separators in series. The lean gas passes from the top of the separators through heat exchangers and into compressors, to be injected back into the formation whilst the condensate is taken from the bottom of the separators. The calcium-chloride circuit is continuous, taking the solution from concentrators, pumping it through the refrigeration plant and into the dehydrator, where it meets the gas from the scrubber and, after dehydrating it, passes through horizontal separators and back into the concentrate tank. A. H. N.

1355.* Exact Technique for Shooting Wells. Anon. *Oil Wkly*, 12.8.40, **98** (10), 30.—Shooting in the Illinois Basin whenever output declines to uneconomical levels has become common practice and an exact technique has been developed.

The first step is to remove the pumping unit and clean the hole by means of a small portable drilling machine. A shot is lowered and set according to accepted practice. A bridge is placed immediately above the time-bomb and the hole is tamped.

Two tamping methods are in common use : the "pea-gravel" tamp, and the other involving the use of quick-setting cement. These two methods are detailed. The force of the explosion is expended horizontally into the formation, and not upwards injuring the casing. The top of the casing is left open in both cases.

Clean-out methods are described for removing the debris from the bottom of the well. Ordinarily the time taken for preconditioning the well, cleaning out, and replacing the tubing and rods is 7 days. A. H. N.

1356.* Removing Paraffin from Pipe and Sand Face with Carbide. F. R. Cozzens. *Oil Wkly*, 19.8.40, **98** (11), 25.—The treatment of the sand face with carbide consists of removing the tubing and rods, bailing the hole as dry as possible, and then filling with water. A nipple or section of pipe about 8 ft. long, open at the top, with a slot cut in the side, is screwed into the casing-head to permit the escape of carbide fumes above the worker's head.

Carbide, in ¼ × 1-in. pieces, is rapidly dumped down the casing through the slot in the fume-pipe, 100 lb. carbide/brl. of water in the reservoir. The heat of reaction between water and carbide is sufficient to raise the temperature of the water to 100–125° F. After the well has stood for an hour, the fume-nipple is removed and the shot-hole bailed out while the residue, which contains the melted paraffin, is still hot.

The action of the carbide also results in caving of the sand-body, and when the accumulation is removed a fresh sand-face is provided in the well. A. H. N.

1357.* Distillate Fields Bring New Production Phase. Part IV. B. Mills. *Oil Wkly*, 19.8.40, **98** (11), 28.—Recycling plants are considered an economic necessity in distillate fields if (1) gas wastage is to be avoided by returning unmarketable stripped residue to the producing formation, and (2) reservoir pressure is to be maintained so as to prevent the occurrence of retrograde condensation and subsequent loss of recoverable contents.

Of the several types of recycling plants, this paper discusses some of those operating on the absorption principles. An interesting development in the adaptation of the absorption method in recycling operations has been the remarkable increases found feasible in absorption pressures. Whereas a short time ago it was not considered desirable to attempt absorption at pressures greater than 800–900 lb./sq. in., modern recycling plants are operating at pressures up to 1500 lb./sq. in., and tests indicate that absorption pressures in excess of 2000 lb./sq. in. are practical.

Typical absorption-type recycling plants are illustrated and discussed individually, together with details of dimensions, capacities, and characteristics. A. H. N.

1358.* Butane Used as Penetrant in Acidizing Kansas Completion. Anon. *Oil Wkly*, 19.8.40, **98** (11), 46.—A production test on a newly drilled well showed that the well yielded 28 brl. in 2 hr. The crude was heavy, 20° gravity A.P.I. Study of the problem indicated that the crude was of such a consistency that it might insulate the acid from the formation ; it was consequently decided to use a diluent, in order to reduce the viscosity of the crude immediately near the sand face.

A compounded method was applied in stages. As a first step an acid gun cleaned the sand face with an acid jet. Compounded pumps then forced butane into the sand through a tubing string, the annulus between tubing and casing being filled with water. Finally, acid was forced into the formation.

The results obtained were satisfactory, although the effects of the butane cannot be individually assessed. A. H. N.

1359.* Practical Advantages of Small Area Pressure Maintenance. Anon. *Oil Wkly,* 26.8.40, **98** (12), 18–22.—After development was substantially complete, operators owning all but a small amount of extreme-edge leases formed a unit to operate the Wilcox sand production of the pool. This made it possible to initiate careful reservoir control and to take advantage of a pronounced and efficient water drive. The sand has zones of varying permeability, and in order to prevent an uneven water encroachment and to prolong the flowing life of the wells, the rate of withdrawal was voluntarily reduced and pressure maintenance instituted.

The bottom-hole pressure has been maintained within 93% of its original status, although 7½ million barrels of oil have been produced. The decline has been practically eliminated in recent months, indicating a long flowing life for the field. Although the field covers scarcely more than 1 sq. mile and was substantially developed when unit operation was inaugurated, scientific exploration is estimated to have effected a saving of $500,000 by the elimination of unneeded wells and prolongation of flowing life. It is further predicted that proper utilization of reservoir energy will reduce operating costs by an estimated $1,500,000. The lifting cost has been only a fraction more than 3 cents/brl.

Details of the operations and practices are given. A. H. N.

1360.* Distillate Fields Bring New Production Phase. Part 5. B. Mills. *Oil Wkly,* 26.8.40, **98** (12), 26.—This forms the concluding part of the symposium on recycling operations, and continues the discussion of absorption-type distillate recycling plants commenced in Part 4. It is a description of a series of typical plants, giving photographic illustration and individual details of the dimensions, capacities, and characteristics of each. A. H. N.

1361.* Gas and Oil Separation in Deep Fields of California. W. A. Sawdon. *Petrol. Engr,* August 1940, **11** (12), 42.—In general, two methods are employed: two-stage separation at the well, and divided separation with high-stage separation at the well and low-stage separation at a central station. Both methods are described and illustrated.

Except for a new trap in which tests have recently been completed, the high-pressure separators have been capable of handling production at the rate of 1500 brl./day. The new type can handle 2500 brl./day.

Gas from each separator, the high- and low-pressure, is measured by orifice meters, and the recent practice has been to place the orifice flange on the vertical gas line leading downward from the top of the separator. This eliminates the collection of condensate at and around the orifice and has been found to be a very satisfactory arrangement.

Where the second method is used, the oil arriving at the central station from the well passes through a low-pressure separator, where the remainder of the gas is removed. This gas is then recompressed and enters the absorber with the high-pressure gas. At low production rates 80% of the gas is separated under high pressures. A. H. N.

1362.* Deepening and Completing a Well in the Lisbon Field, Louisiana. Part 7. P. D. Torrey and F. H. Miller. *Petrol. Engr,* August 1940, **11** (12), 63.—This paper gives further details of production tests and analysis of the crude oil.

A discussion is given on the estimation of oil and gas reserves in a new field as illustrated by the typical case under study. Three types of estimates are made, the method of calculation being presented in a table. The first estimate presupposes using pressure maintenance, gasoline extraction, and optimum separation pressures. The second estimate differs from the first in using 2000 lb./sq. in. at 65° F. for separation. The third method does not include a gasoline plant, but is otherwise similar to the second.

Finally, a table gives estimated recoveries and gross value of production from different well-spacing units for each of the three methods. The units for well spacing are 160-acre, 320-acre, and 640-acre, respectively. Under all three methods of production the total gross value of gas and condensate was a maximum for the 640-acre spacing. Of the three methods, the first yielded the highest value. A. H. N.

1363.* Proper Sealing of Abandoned Wells Important in Mining Areas. F. R. Cozzens. *Petrol. Engr*, August 1940, **11** (12), 104.—The importance of proper sealing of abandoned wells so that gas cannot travel through porous formations into a coal seam, and hence to the mines and be a source of dangerous explosions, is emphasized.

One device generally used is a tapered plug of wood, driven into the sand, and backed-up with cement slurry. This method is effective if sufficient loose material falls on top of the cement to close the cavities which form in the walls as the casing is lifted. Otherwise gas pressure increases, and over a course of years becomes sufficient to force openings around the weakened cement seal. Closing the hole at the surface tends only to increase the pressure below ground.

It has been found that a filler of residue placed over a plug is more vital to a permanent seal than the type of plug used. The filler must be resilient enough to fill cavities, yet solid enough to pack into a continuous gas-proof mass. Silt or fine pulverized dry clay is suitable.

Even more effective is the procedure of mixing the silt or the fine clay with coarse salt in a ratio of 1 lb. salt to 10 lb. silt. The mixture is dumped dry, and no water is used. Moisture is drawn from the walls, and the pack settles quickly and effectively. A. H. N.

1364.* First Six Months' Production (in U.S.A.). W. V. Howard. *Oil Gas J.*, 25.7.40, **39** (11), 78–79.—Crude-oil production for the first six months of 1940 was 684,733,094 brl. This represents 11·1% increase over the first half of 1939 and an increase of 10·6% over the corresponding period of the peak year of 1937. Average daily production of 3,762,270 brl. is 56·5% greater than 1933, and by far the greatest part of this increase is due to a rise in domestic demands. Of the nation's supply, 58·8% or 404,400,039 brl. came from seventy-five fields, of which East Texas produced 71,856,874 brl. A. H. N.

1365.* Cotton Valley Agreement Offers Complete Pressure Maintenance. G. Weber. *Oil Gas J.*, 25.7.40, **39** (11), 168.—Oil operators and landowners of the Cotton Valley field have unitized all their holdings in that field to set the stage for the beginning of the largest unitized recycling project ever undertaken. This agreement establishes a precedent and furnishes an example for the formulation of similar, much-needed instruments in other condensate fields. The paper discusses the significance of the terms of major importance in the agreement. A. H. N.

1366.* Less Heat, More Chemical May Increase Profits. T. P. Sanders. *Oil Gas J.*, 25.7.40, **39** (11), 174.—Many oil companies, in accounting treating costs, do not place sufficient emphasis on losses in oil gravity resulting from breaking the emulsion, but give much prominence to expenditure for treating chemicals. Oftentimes a study will reveal that an overall saving may be effected by the use of more chemicals and less heat, or by proper arrangement of lease batteries. A. H. N.

1367.* Systematic Study of Reservoirs for Efficient Pool Operation. T. P. Sanders. *Oil Gas J.*, 1.8.40, **39** (12), 30–32.—Most oil companies were originally organized with a rather sharp division between producing and geological departments. While results show that such a form of organization has been eminently successful, in many instances it has been found that this limits the use of practical geology and geological records in solving producing problems. For overcoming this objection several different forms of organization have proved successful.

One such organization is undertaken by a pool-study department within the producing department. It is the duty of this group to combine all geologic and production data in order to present a coherent picture of the reservoir as it exists at various stages during its productive life.

The data required for a pool-study is tabulated as: (1) Elevation. (2) Log: casing; depth of showings; water encountered; top of producing horizon; penetration; bottom of producing horizon; shot or acid record; total depth. (3) Initial shut-in casing pressure. (4) Initial production or potential for both oil and gas. (5) Fluid levels, static, oil and water. (6) Continuous record of production of oil, gas, and water. Gas–oil ratio and water % from periodic tests. (7) Complete operating history, showing plug-backs, reperforating, shooting, acidizing, giving dates, depths, and production before and after.

For new pool-studies the following additiona records are recommended: (1) Electric log. (2) Core description. (3) Core analysis. (4) Bottom-hole pressures: (a) as early as possible after discovery; (b) at regular monthly intervals. (5) Bottom-hole fluid samples, initially and also later. (6) Productivity tests. A. H. N.

1368.* Economical Pumping of Wells. T. P. Sanders. *Oil Gas J.*, 15.8.40, **39** (14), 48.—In the field under study it was predicted that pumping would prove uneconomical at the 6500 ft. depth due to sand troubles, gas lock, and water encroachment. Almost all the wells, however, are now pumped economically, and many of them for a small daily production. The practices adopted and servicing methods used are described in detail.

Sand troubles are overcome by using screens and prepacked gravel liners. The characteristics of the gravel packs are given. Another method adopted is the use of a "sand anchor"—a sand screen which somewhat resembles a gas anchor. Metal-to-metal pumps are used, but in one place a plunger is fitted with combination rubber and fabric rings mounted in machined grooves and dipped in kerosene in order to cause it to expand and fit snugly in the barrel. The ends of the plunger never emerge from the barrel during pumping.

To combat rod abrasion by the sand, rod rotators are installed on the beam, thus distributing the wear over the surface of the rod. The rods are suspended from a swivel, and a wire line hitched on to the walking beam operates a ratchet on a notched disc, thereby turning the rods several degrees. A. H. N.

1369.* 2,930 Ft. of Welded Casing Run in Well in Seven Hours. H. F. Simons. *Oil Gas J.*, 15.8.40, **39** (14), 61.—Dimensional details of the casing joints used are given. Time of running casing is reduced by 33·3% by using long joints, thus reducing the number of welds.

One of the savings resulting from using this type of running casing is due to the fact that only two welders are needed in addition to the regularly employed crew, instead of three men to help the regular crew in ordinary jobs. The welded casing requires neither spinning up nor tonging, and hence the regular crew can perform all the work but the welding.

After the weld is finished, it has to be left to cool for a minimum of 45 secs. before lowering it into the hole. This procedure allows the weld to cool to a temperature below the critical point. Lowering the pipe into the hole sooner than this would result in its being prematurely quenched in the drilling mud, thereby weakening the structure of the welded portion and making failure possible. Average time to run a joint of the casing is 4½ min., including time for picking up the joint, inserting it, welding and lowering the casing ready to pick up the next joint. A. H. N.

1370.* Intermittent Gas Lift Beneficial to Large Wells. T. P. Sanders. *Oil Gas J.*, 15.8.40, **39** (14), 63.—It is pointed out that intermittent gas lift is capable of reducing the gas–fluid ratio on almost any well which produces less than 1000 brl./day of fluid.

This very short paper is based on the data presented by E. C. Babson before the American Petroleum Institute. (Cf. abstracts Nos. 263, 265, 267, and 271.)

1371.* Production at the Oilfield El Trebol. F. R. Ruiz. *Bol. Inform. Petroleras*, April 1940, XVII (188), 33–42.—A gradual fall of pressure in this oilfield called for special measures in order to maintain satisfactory output. The article describes in detail, with curves, the decline in pressure, deals with the pumping in of gas, methods of drying the latter, its solubility in the petroleum, etc., and gives tables of pressures and quantities. Apparently production was unduly forced in some of the wells, owing to too optimistic expectations. H. I. L.

1372. Patents on Production. De M. G. Miller. U.S.P. 2,209,466, 30.7.40. Appl. 13.12.38. Seal indicator for pumps of the deep-well turbine-pumps type.

T. V. Moore. U.S.P. 2,209,534, 30.7.40. Appl. 6.10.37. Method for producing gas wells by adisbatic or polytropic expansion of the high-pressure gas into a chamber and repassing the cooled low-pressure gas through a heat exchanger to cool the high-pressure gas to its dew point and separating the liquid.

L. C. Miller. U.S.P. 2,209,627, 30.7.40. Appl. 27.3.39. Well device and operating means therefor, for closing a well utilizing fluid pressure.

C. P. Walker. U.S.P. 2,209,944, 30.7.40. Appl. 7.9.37. Method of measuring location of obstructions in deep wells by means of the echo recordings of a pressure wave generated in the annulus between the tubing and casing.

E. F. Raymond and L. Spencer. U.S.P. 2,210,125, 6.8.40. Appl. 1.11.37. Gun perforator for well casings, consisting of a wire-rope connection, loosening jar structure to give hammering blows, and a firing mechanism.

G. F. Turechek. U.S.P. 2,210,192, 6.8.40. Appl. 13.3.39. Gun-perforator socket for receiving bullet and explosive charge.

R. D. Elliott. U.S.P. 2,210,205, 6.8.40. Appl. 30.3.39. Water-intrusion location in oil wells characterized by injecting an oxidizing agent into the region under test and measuring polarization effects by means of an electrode. Agent becomes diluted by incoming fluid.

N. R. Kimmel. U.S.P. 2,210,245, 6.8.40. Appl. 27.9.38. Formation tester.

R. R. Kyner. U.S.P. 2,210,247, 6.8.40. Appl. 13.11.37. Gas-lift apparatus.

M. O. Boulter. U.S.P. 2,210,359, 6.8.40. Appl. 12.9.39. Well-tubing paraffin remover, the tool being driven hydraulically downwards.

M. M. Kinley. U.S.P. 2,210,417, 6.8.40. Appl. 1.11.37. Leak detector by altering height of fluid in casing and picking up sound generated by flow of liquid, the intensity being an indicator of depth of leak.

A. C. Hamilton, Jr. U.S.P. 2,210,545, 6.8.40. Appl. 15.11.39. Process of sealing openings in the earth.

K. Grosse and G. Schlicht. U.S.P. 2,210,582, 6.8.40. Appl. 12.9.38. Method for the extraction of petroleum by mining operations after degassing the formations through a vertical shaft, by using horizontal and inclined drift network system.

C. C. Christian. U.S.P. 2,210,885, 13.8.40. Appl. 30.4.36. Well construction using perforated casing.

A. Hollander. U.S.P. 2,210,977, 13.8.40. Appl. 8.12.37. Strainer for submersible motor pumps adapted to be connected between motor and pump.

J. W. MacClatchie. U.S.P. 2,210,983, 13.8.40. Appl. 13.4.37. Method and means for perforating well-casing in place by means of a gun fitted with a safety mechanism, which gun is lowered on a cable and can be fired at a number of points.

J. W. MacClatchie. U.S.P. 2,210,984, 13.8.40. Appl. 23.12.38. Means for perforating well-casing consisting of a gun with a multiple cartridge magazine and multiple firing mechanism.

J. H. Howard. U.S.P. 2,211,122, 13.8.40. Appl. 10.3.38. Tubing head and hanger with a multiplicity of sealing means.

J. M. Pearson. U.S.P. 2,211,158, 13.8.40. Appl. 13.9.38. Method for orienting cores using magnetic properties.

J. H. Howard. U.S.P. 2,211,206, 13.8.40. Appl. 13.3.37. Method and apparatus for completing a well where it is required to run a screen into a high-pressure well having a casing closed near its upper end.

G. W. Haury. U.S.P. 2,211,776, 20.8.40. Appl. 2.8.37. Resilient coupling for tubing and method for making the same.

C. C. Brown. U.S.P. 2,211,845, 20.8.40. Appl. 9.11.33. Means for equipping wells under pressure.

C. C. Brown. U.S.P. 2,211,846, 20.8.40. Appl. 26.11.37. Valve of the sleeve type in a tubing.

C. R. Athy. U.S.P. 2,211,965, 20.8.40. Appl. 9.11.36. Chain mechanism for connecting pump-rods with walking beams.

C. W. Parris. U.S.P. 2,211,983, 20.8.40. Appl. 4.11.37. High-pressure joint for tubes and the like.

H. C. Otis. U.S.P. 2,212,036, 20.8.40. Appl. 3.4.37. Union for well-heads in combination with an oil-saver.

R. M. Ridley. U.S.P. 2,212,044, 20.8.40. Appl. 24.7.39. Method and apparatus for perforating well-casings.

R. M. Ridley. U.S.P. 2,212,045. 20.8.40. Appl. 24.7.39. Gun perforator.

J. F. Eaton and H. D. Collins. U.S.P. 2,212,153, 20.8.40. Appl. 10.2.38. Vibration damper to be incorporated in sucker-rod strings.

A. L. Parker. U.S.P. 2,212,183, 20.8.40. Appl. 2.3.38. Tube coupling.

W. N. Eddins. U.S.P. 2,212,416, 20.8.40. Appl. 21.2.38. Fluid-operated pump for deep wells.

A. H. N.

Natural Gas.

1373.* Gas Dehumidification Prevents Hydrate Freezing Troubles. E. G. Hammerschmidt. *Nat. Petrol. News*, 15.5.40, **32** (20), R. 162.—Various methods of eliminating hydrate freezing troubles in natural gas and natural gasoline plants are discussed. In general all the methods remove water condensate and sufficient water vapour from the system so that further condensation cannot occur either under conditions of minimum temperature or maximum pressure. Seven gas-washing solutions are described. These include calcium chloride, diethylene glycol, and lithium chloride. The last-named has been used only on air-conditioning plants, but since it offers many advantages over $CaCl_2$ its extended use is to be expected. Glycerine is sometimes used in conjunction with $CaCl_2$. Solid adsorbants simplify plant design owing to their smaller relative volume. Silica gel, activated alumina, anhydrous calcium sulphate, and bauxite impregnated with manganous chloride are all in successful use.

H. G.

1374.* Manufacture of *iso*Butane at Kettleman Absorption Plant. L. P. Stockman. *Oil Gas J.*, 15.8.40, **39** (14), 50.—A description is given of a recently modernized Kettleman Hills gasoline-absorption plant, originally designed to handle 107 million cu. ft. of gas, and to produce 125,000 gal. per day of finished gasoline, and now modified to produce *iso*butane. The original stabilizer column has been converted into a depropanizer, and new debutanizer and de*iso*butanizer columns have been added. A propane refrigeration unit operating under automatic pressure control maintains temperature conditions on the absorption oil and wet gas. Positive cooling for the propane condensate and reflux is provided by a simple unit forced-draft cooling-tower operating in conjunction with the atmospheric tower. An increased production of 6% of gasoline has resulted, in addition to yields of other hydrocarbons, in a relatively pure state. Simplified control, reduction of fluctuation, and enhanced value of the gas have also resulted. *n*-Butane of purity ± 98% and *iso*butane of purity 96–98% containing 2–2½% of *n*-butane and a trace of propane are produced. A flow-sheet and operating data of the plant are presented. C. L. G.

Cracking.

1375.* Houdry Units' Heat-Transfer Salts Described at A.I.C.E. Meeting. Anon. *Nat. Petrol. News*, 12.6.40, **32** (24), R. 220. (*Abstract of paper read by W. E. Kirst, W. M. Thayle, and J. B. Castner vide A.I.C.E. Transactions, Vol.* 36, *No.* 3, *June* 1940.)—Molten salts consisting of 40% $NaNO_2$, 7% $NaNO_3$, and 53% KNO_3 are used on a large scale on Houdry cracking units. They possess a low melting point, high heat-transfer rate, and adequate thermal stability, and are applicable at a higher temperature than any other commercial heat-transfer medium without recourse to elevated pressure. Corrosion problems are claimed to be not great. Open-hearth steel may be used up to temperatures of 850° F. High-chromium and certain low-chromium–aluminium–silicon steels are recommended for temperatures up to 1100° F. H. G.

Hydrogenation.

1376. Reactions of the Catalytic High Pressure Hydrogenation of Coal and Oil and Their Control. M. Pier. *Trans. Faraday Soc.*, 1939, **35**, 967–979.—An account is given of the main principles of high-pressure hydrogenation. Two processes are general; liquid-phase treatment with low catalyst concentration, and vapour-phase hydrogenation with high catalyst concentration. Sometimes necessary also are a refining process to remove O_2, S and N, and an ash-removal process. Temperatures are usually below 400° C., although in a few cases high anti-knock petrols can be obtained at 500° C. Recycling of products is universal.

An analysis of various starting materials and catalysts is given, together with the anti-knock values of the petrols obtained. The author concludes that by this process all types of hydrocarbon oils, from high-quality aviation spirits to lubricating oils, can be obtained by suitable choice of raw materials and catalyst. M. McL.

1377. Patent on Hydrogenation. G. W. Johnson. E.P. 524,380, 6.8.40. Appl. 8.4.38. Catalytic conversion of hydrocarbons containing more than 15 gm. of hydrogen to 100 gm. of carbon by subjecting to cracking or hydrogenation under pressure and in the presence of an initially highly active catalyst, but the activity of which has been reduced by treatment of substances poor in hydrogen. H. B. M.

Polymerization.

1378.* Sohio Polymerization Unit First Using New Catalyst. M. G. Van Voorhis. *Nat. Petrol. News*, 26.6.40, **32** (26), R. 230.—Copper pyrophosphate is the catalyst used in the plant described. The feed, from high-pressure stabilizers, contains 32·4% unsaturates which suffer 80% conversion into 84 O.N. polymer gasoline. At least 95% of the product boils below 400° F. and it is all blended into motor fuel. The catalyst is not recovered, but is discarded when spent. H. G.

1379. Patents on Polymerization. Universal Oil Products Company. E.P. 524,986, 20.8.40. Appl. 10.1.39. Treatment of hydrocarbon mixtures containing 3 or 4 carbon atom olefins and derived from the cracking of petroleum oils to render them suitable for catalytic polymerization without deleterious effects on the catalysts employed. The olefin-containing mixture is washed in liquid phase with water for the removal of basic impurities and with aqueous alkali for the removal of acidic impurities.

The Distillers Co., Ltd. E.P. 525,276, 26.8.40. Appl. 16.2.39. Depolymerization of polystyrene by subjecting it either in pure or crude form to rapid heating of short duration, the speed of which is ensured by intimately contacting the polystyrene with the body or surface of a heated material of high heat conductivity at a temperature between 550° and 700° C. H. B. M.

Alkylation.

1380.* Neohexane Plant Operating at Record High Pressures. R. C. Alden. *Nat. Petrol. News*, 26.6.40, **32** (26), R. 234.—A large-scale plant for the production of neohexane from ethylene and *iso*butane is described. *iso*Butane at 4500 lb./sq. in. is pumped through an alkylation coil at 950° F. Controlled quantities of a mixture of ethylene and *iso*butane are admitted to the coil at several points. The products are neohexane and high-octane-number gasoline, the latter containing appreciable quantities of *iso*pentane resulting from the interaction of ethylene and propane. The boiling point of neohexane is sufficiently different from those of the hydrocarbons occurring with it to ensure its removal, by fractionation, in a reasonably pure state. Since neohexane is a pure hydrocarbon, the commercial product has a more constant octane number than that of commercial *iso*-octane, which is usually a mixture of 2 : 2-dimethylbutane and 2 : 2 : 4-trimethylpentane. The high octane number, high lead susceptibility, and high volatility of neohexane will overcome the volatility obstacle to the development of aviation fuels with octane ratings above 100. In addition to neohexane, the plant described offers facilities for the large-scale manufacture of *iso*pentane by separation from *iso*-octane and for the preparation of straight-run gasoline and natural gasoline. A range of gasolines of octane number up to 115 is therefore available. H. G.

1381.* Alkylation Plant takes Olefins left by Selective Polymerization. A. H. Garrison. *Nat. Petrol. News*, 12.6.40, **32** (24), R. 212.—An outstanding development of the past year in the utilization of light petroleum fractions to produce high-grade aviation fuels has been the rapid expansion of the sulphuric-acid alkylation process. In the plant described the charging stock consists of the residual butane–butene fraction from hot acid polymerization. Substantially all the olefinic portion of the charge consists of the normal butenes remaining in the residual C. 4's after the removal of all the *iso*butene and dienes and part of the normal butenes by the hot-acid process. Alkylation is carried out at 40–60° F. with 96–100% H_2SO_4. The major heat load is carried by exhaust steam, and refrigeration is by means of an ammonia absorption-type unit. No very unusual control problems arise except in the de-*iso*butanizer column, which operates at a 20° temperature differential from top to bottom. Acid consumption is at the rate of 0·55–0·6 lb. per gal. of alkylate. The product has a Reid vapour pressure of 2·5 lb. and contains 0·001% S. Its end-point is 375° F., with 90% recovery at 255° F. H. G.

Synthetic Products.

1382. Petroleum Synthesis can Supply Rubber in National Emergency. J. Farquar. *World Petrol.*, August 1940, **11** (8), 64.—The development, production, and properties of synthetic rubber, particularly of the Buna type, are discussed, and reference is made to the possibility of supplying the entire U.S. requirements with synthetic rubber manufactured from petroleum. The U.S. consumes 600,000 tons of rubber per annum (60% of the world's rubber supply), importing this from Ceylon and the East Indies. Up to six months' supply is available in storage, but in the event of emergency it would be possible to complete sufficient industrial plants to make synthetic rubber, at a cost of perhaps $100,000,000. Production of neoprene and thiokol amounts to 1750 tons per annum, but is expected to increase to 4000 tons per annum by the end of the year. German production of Buna rubber from coal provides half their requirements, and this is expected to increase to three-quarters. A plant is being constructed at Baton Rouge to produce 5 tons of synthetic rubber per day, at a cost of $1,000,000–$1,250,000, by the end of 1940.

Synthetic rubber in Germany consists of Buna S from butadiene and styrene and Buna N (or Perbunan) from butadiene and acrylo-nitrile. The former has a greater resistance to abrasion than natural rubber, and the latter, in addition, is superior in resistance to deterioration in heat, and oil, and in ageing qualities. Perbunan Extra (Buna NN) is a copolymer of butadiene with a higher percentage of acrylo-nitrile. The greater toughness of synthetic rubber over natural rubber is attributed to the cross linkages in the molecule formed during polymerization, whereas the rubber molecule consists largely of straight-chain polymers. This is particularly the case with the Buna S, but less so in the case of the butadiene–acrylo-nitrile polymers which are formed in an emulsion. This cross bridging results in some difficulty in mastication on the rubber mill, so that it is necessary to add softeners. Less sulphur is, however, required for vulcanization. Perbunan tends to harden on heating, becoming stiffer on ageing.

The improved resistance to swelling in oil and other solvents is believed to result from the presence of the nitrile groups in the polymer. The low tensile strength of the pure Perbunan (800 lb./sq. in.) is greatly increased by the addition of 40–50% of channel black (to 4000–5000 lb. per sq. in.). A similar proportional increase in the strength of pure rubber (3500 lb. per sq. in.) on reinforcing would yield a remarkable structural material, so that research along these lines is indicated.

Perbunan is expected to be produced at a cost of $0·20 to $0·30, comparing favourably with neoprene at $0·75 per lb. and approaching the minimum price of rubber—$0·10 per lb.

Graphical data on the swelling and tensile strength of Perbunan as compared with rubber after immersion in various solvents, and the effect on the tensile strength of Perbunan after the addition of gas-black, are presented. C. L. G.

1383. Patents on Synthetic Products. Armour & Co. E.P. 525,184, 22.8.40. Appl. 17.2.39. Preparation of condensation products by reacting acetylene or an acetylene hydrocarbon with phosgene in the absence of free oxygen.

Les Usines de Mello. E.P. 525,198, 23.8.40. Appl. 15.2.39. Preparation of diolefines by the catalytic dehydration of the corresponding glycols, olefinic alcohols, or mixtures of these compounds. Volatile phosphorous-containing compounds are used as catalysts. H. B. M.

Refining and Refinery Plant.

1384.* Desulphurization of Ishimbaevo Gasoline with Hypochlorite. B. A. Englin, *Neft. Khoz.*, 1939, No. 8, 36.—It has been found that gasolines from Ishimbaevo crude, which do not contain hydrogen sulphide, can be desulphurized under refinery conditions by acidification with hypochlorite to an acid reaction. Under suitable conditions of treatment and right commencement of acidification a considerable improvement is achieved. If 1·5% (4%) hypochlorite, taken as dry calcium hypochlorite, is added, a gasoline is obtained which, after redistillation with 95% yield, contains 0·155% (0·105%) sulphur and 0·017% (0·025%) chlorine, respectively. With higher percentages of hypochlorite, sulphur can be entirely eliminated.

The treatment is as follows: gasoline from the rectifying column flows into the mixing condensers, where the hydrogen sulphide and lower mercaptans are removed by alkali wash. Thereupon the gasoline is treated with hypochlorite in two stages. First it is mixed with alkaline hypochlorite to a negative doctor test. During the whole of the following process $\frac{1}{2}$–1*N*-sulphuric acid is added. The hypochlorite consumption is in the first stage 1·6–2% and in the second 1·8–2·2%, that of the sulphuric acid 0·04–0·06% and 0·1–0·13%, respectively. The second batch of hypochlorite is added after decantation of the first. When the hypochlorite treatment is finished, the gasoline is washed with water and redistilled with alkali. A 2·5% alkali–gasoline solution is supplied into the still, where it is mixed with superheated steam of 200–250° C. The gasoline yield is 97–98%. As an emergency, distillation may be carried out without alkali, but the output will then be only 95%, with a subsequent alkali and water wash. L. R.

1385.* Italians Process Albanian Heavy Oil to Aviation Gasoline. E. A. Bell. *Oil Wkly*, 22.7.40, 98 (7), 28.—In 1868 an oil zone was discovered north-east of Valona, and the development which followed gave an annual crude output which averaged 6000 tons before 1914. After the war several companies took up concessions, but finally all the areas came under Italian control. Very satisfactory results have been obtained in the Devoli region, and early in 1939 there were 250 wells giving oil from depths of 1640–2620 ft.

The oil is asphaltic and gives 12–18% of gasoline. On cracking it may give a maximum of 40% of gasoline, and on hydrogenation it can give 80% of gasoline of high octane number. The hydrogenation is carried out at Bari and Livorno. Each plant has a capacity of 120,000 tons/year of 100-octane gasoline and 30,000 tons of high-grade lubricating oil. There are other products. These plants can also be used for the hydrogenation of lignite and bituminous shales.

Italy imports the bulk of her oil requirements, and in 1938 her crude oil production, never very large, had fallen to 13,000 tons. G. D. H.

1386.* Gasoline Treating Patents Issued One Weekly in 1939. Anon. *Nat. Petrol. News*, 15.5.40, 32 (20), R. 179.—Brief details are recorded of the relevant patents under four headings: (*a*) Sweetening processes, (*b*) gum stabilization processes, (*c*) anti-knock improvement processes, (*d*) miscellaneous processes. H. G.

1387.* Specification of Materials for Refineries. A. Bertoni. *Bol. Inform. Petroleras*, April 1940, XVII (188), 43–53.—This article discusses the methods adopted in specifying pipes, valves, and accessories required in refinery work. Schedules are given for seventeen different classes of material classified according to the purpose for which they are to be used. The author refers to the I.R.A.M., which lays down the qualities of materials to be used in every case, and he suggests that it would be advantageous if an official Materials Testing Institute could be set up, particularly in view of the war, when certain standard materials may be unobtainable and substitutes would have to be carefully tested. H. I. L.

1388. Patents on Refining. Standard Oil Development Corp. E.P. 523,894, 25.7.40. Appl. 14.10.38. Separation of olefins having 2–5 carbon atoms from mixtures of the same with saturated hydrocarbons. The olefins are absorbed in sulphuric acid and regenerated from the acid liquor. The acid employed is stronger than that normally used, and the acid liquor obtained is heated without dilution in the form of a thin film to a temperature higher than the boiling point of the alcohols that may be formed from the acid liquor.

A. E. Rheineck. U.S.P. 2,210,305, 6.8.40. Appl. 22.11.38. Process for treating an oil from the group consisting of oiticica oil and po-yoak oil to render the oil suitable for use in varnishes, etc. The oil is acetylated until it has the property of drying in a relatively short time and of forming a smooth, glossy film.

R. C. Palmer and C. H. Bibb. U.S.P. 2,211,432. Appl. 19.8.38. Treatment of monocyclic terpene hydrocarbons by heating to reaction temperatures in the liquid phase in the presence of a catalyst containing effective amounts of both copper and nickel in an active form to effect substantially complete conversion of the hydrocarbons into *p*-cymene and *p*-menthane.

Deutsche Erdol, A.-G. E.P. 524,587, 9.8.40. Appl. 1.2.39. Raffination of mineral oils and tars by means of liquids containing phenols. The treatment is effected in steps, the phenol content of the liquid used in a subsequent step being higher than that of the liquid employed in a previous step. H. B. M.

Safety Precautions.

1389.* Prevention of Accidents in the Y.P.F. B. Rikles and R. Galletti. *Bol. Inform. Petroleras*, April 1940, XVII (188), 64–79.—This very fully detailed paper was originally written for the Argentine National Committee of the World Power Conference, and was intended for presentation at the Second Chemical Engineering Congress which was to have met in Berlin in June of this year. The paper should be of considerable interest to those concerned with safety regulations in works generally as apart from oilfields and refineries in particular. H. I. L.

Chemistry and Physics of Petroleum.

1390. Activation of Hydrogen in Catalytic Reactions of Hydrocarbons. A. Farkas. *Trans. Faraday Soc.*, 1939, **35**, 906–917.—The author discusses the formation of hydrocarbon bonds by the activation of hydrogen by metal catalysts. It is shown that hydrogen atoms are formed on the catalyst surface; methods of measurement of the rate of formation are discussed. A possible mechanism for the reactions at high and low temperatures is suggested, and its correlation with practical results investigated. M. McL.

1391. Catalytic Exchange of Hydrogen Atoms between Molecular Deuterium and *n*-Hexane and *cyclo*Hexane. A. Farkas and L. Farkas. *Trans. Faraday Soc.*, 1939, **35**, 917–920.—These exchange reactions are shown to follow a similar mechanism to that proposed for ethylene and benzene (*Trans. Faraday Soc.*, 1937, **33**, 82), involving the formation of hydrogen atoms on the surface of the catalyst. A new mechanism is suggested for the dehydrogenation of *cyclo*hexane. M. McL.

1392. Fundamentals in the Catalytic Ring Closure of Open-Chain Hydrocarbons. H. S. Taylor and J. Turkevitch. *Trans. Faraday Soc.*, 1939, **35**, 921–934.—A general review is made of the thermodynamic and practical problems involved in the large-scale production of aromatics from open-chain hydrocarbons. The use of various catalysts has been investigated, together with the optimum conditions of temperature and pressure; it is found that for paraffin hydrocarbons, long contact time, high temperature, and oxide catalysts, such as Cr_2O_3, are most effective.

The effects of oxygen and water-producing substances in poisoning the catalysts are noted, together with the best methods of conversion of olefines into ring structures. M. McL.

1393. Mechanism of Catalytic Exchange Reactions between Deuterium and Olefines. G. H. Twigg. *Trans. Faraday Soc.*, 1939, **35**, 934–945.—The two possible mechanisms of this interchange are discussed, and experimental work on heavy ethylene, C_2D_4, is described, which indicates that an associative rather than a dissociative mechanism is operative.

Data are given also of energies of activation, of exchange, and of hydrogenation for ethylene, propylene, butene-2, and *iso*butene over the range 55–120° C. M. McL.

1394. Fischer–Tropsch Synthesis of Hydrocarbons, and Some Related Reactions. S. R. Craxford. *Trans. Faraday Soc.*, 1939, **35**, 946–958.—This paper describes a comprehensive investigation into the Fischer–Tropsch synthesis of hydrocarbons as regards optimum conditions and the mechanism of the catalysed reaction. In all cases the standard catalyst, cobalt–thoria–kieselguhr, was used; best results were obtained at medium pressures and in the temperature range 175–225° C.

It is concluded that the mechanism involves the formation of a cobalt carbide Co_2C by a dual process. This carbide is then reduced by hydrogen to give chemisorbed methylene groups, which in turn build up to hydrocarbon chains.

Experimental work included the use of the *ortho-para*-hydrogen conversion as an indication for the reaction, and is described in some detail.

Notes are also made of the condition where the Fischer–Tropsch synthesis fails, forming methane rather than hydrocarbon chains; a suggested explanation is given. The effects of the presence of ethylene are also discussed. M. McL.

1395. Experiments on the Fischer–Tropsch Synthesis of Hydrocarbon from Carbon Monoxide and Hydrogen. E. F. G. Herington and L. A. Woodward. *Trans. Faraday Soc.*, 1939, **35**, 958–967.—The double effect of the Fischer–Tropsch synthesis, in giving either oil and water, or methane and water, has been investigated. The latter effect is found to be due to high-temperature effects on the catalyst, either by insufficient initial cooling or too rapid subsequent heating. The methane production is also promoted by a high H_2/CO ratio in the ingoing gas.

A "running-in" process for the catalyst is described, which ensures the production of oil.

Further effects are described as follows: the use of hydrogen at synthesis temperature on a used catalyst, causing immediate revivification of the latter and the complete poisoning of the catalyst by excess H_2S or CS_2 at 200° C. A suggested explanation of the former effect is given. M. McL.

1396. Mechanism of Catalytic Dehydrogenation and Cyclization. R. C. Pitkethly and H. Steiner. *Trans. Faraday Soc.*, 1939, **35**, 979–984.—Investigating the mechanism of ring formation by cyclization of paraffins, the authors, using *n*-heptane, have obtained heptene and toluene. No *cyclo*-paraffins, *cyclo*-olefines, nor di- or tri-olefines were to be detected. Experiments were made to show whether the heptene was a by-product or an intermediate in the reaction. Measurement of equilibrium partial pressures indicated that it was an intermediate—a fact supported by thermodynamical reasoning. Two possible mechanisms of the reaction are discussed. M. McL.

1397. Destructive Hydrogenation and Destruction of Hexadecane. H. I. Waterman and J. J. Leendertse. *Trans. Faraday Soc.*, 1939, **35**, 985–992.—The destructive hydrogenation and cracking of hexadecane at 435° C. for 1 hr. using nickel–kieselguhr and chromium oxide catalysts have been studied.

Destructive hydrogenation gave practically no products of molecular weight higher than that of the starting material, indicating an almost complete absence of side-reactions. The products were normal or nearly normal paraffins.

Cracking in the presence of nitrogen gave cyclic and branched hydrocarbon products, including appreciable quantities of hydrocarbons of higher molecular weight than hexadecane; indicating the occurrence of polymerization of primary unsaturated reaction products. M. McL.

1398. Investigation with the Cyclization (Aromatization) of Aliphatic Hydrocarbons. H. Hoog, J. Verheus, and F. J. Zuiderweg. *Trans. Faraday Soc.*, 1939, **35**, 993–1012.—In this investigation many aliphatic hydrocarbons were passed over Cr_2O_3 catalyst at 465° C., atmospheric pressure and 17 ml./hr./55 gm. catalyst rate of flow. Yields and compositions of products were determined.

The main conclusions were: hydrocarbons the structure of which permits of direct formation of a 6-membered ring will be cyclized to a marked extent, others will not. Side reactions are shown by individual groups; thus paraffins are dehydrogenated to aliphatic mono-olefines, aliphatic olefines are either hydrogenated to paraffins or undergo a shift of the double bond, *cyclo*-olefines are hydrogenated to naphthenes, naphthenes show no side reactions.

Paraffins, and olefines with 8 C-atoms, show some cracking reactions. The degree of aromatization increases in the order paraffins, aliphatic olefines, 6-ring naphthenes, 6-ring *cyclo*-olefines, for corresponding compounds.

Suggestions are given for the mechanisms involved. M. McL.

1399. Some Problems Associated with Olefine *iso*Paraffin Addition Reaction. S. F. Birch and A. E. Dunstan. *Trans. Faraday Soc.*, 1939, **35**, 1013–1021.—These authors discuss the addition of olefines to paraffins to give higher paraffins, in the presence of sulphuric acid at low temperature. The products are a complex mixture of *iso*-paraffins b.pt.'s 27–185° C., together with a small olefinic residue. It is shown that the action of the acid is not purely catalytic, a highly unsaturated oil being recoverable by dilution of used acid.

A mechanism is suggested of proton addition from acid to olefine, followed by action between the activated olefine, and an acid paraffin polar complex. The *iso*paraffins would then be formed by a series of side reactions. M. McL.

1400. Heavy Metal Sulphides as Catalysts for the Polymerization of Olefine Hydrocarbons. E. K. Ingold and A. Wasserman. *Trans. Faraday Soc.*, 1939, **35**, 1022–1024.—The use of heavy metal sulphides as catalysts in the polymerization of simple olefines was investigated. It was found that the white and yellow sulphides were inactive, but that the black sulphides, particularly cupric sulphide, were more effective. The most effective agent was found to be cupric sulphide which had been allowed to stand in the air until 30% cupric sulphate had been formed. The sulphide had to be prepared from $CuSO_4 + H_2S$ in acid solution.

A suggested mechanism for this catalysis in the case of the polymerization of β-butylene is given. M. McL.

1401. Dimerization Reactions of Unsaturated Hydrocarbons. E. Bergmann. *Trans. Faraday Soc.*, 1939, **35**, 1025–1034.—This is a general review of the nature of dimerization as applied to various heights of unsaturated hydrocarbons. Two main types of dimerization occur: "Dimerization proper," with formation of *cyclo*butane derivatives, an action which only occurs by irradiation, and "migratory" dimerization with formation of a new olefine caused by migration of a hydrogen atom. This latter alone can be considered as the elementary stage of polymerization. Reviewing the experimental work, the author finds that for mono-olefines and dienes the mechanism of polymerization cannot be made to correspond at all with that of "migratory" dimerization; so that no general rules can be laid down for these classes. For acetylenes and for triphenylallene "migratory" dimerization can be satisfactorily applied, although in the latter case it is followed by *cyclo*-isomerization of the acylated allene derivatives. M. McL.

1402. Low-Molecular Polymerization of Dienes and Trienes. E. H. Farmer. *Trans. Faraday Soc.*, 1939, **35**, 1034–1040.—This deals with the polymerization of conjugated and unconjugated di- and tri-enes. It is found in general that two forms of polymer occur: linear polymers of large molecular weight and low molecular series of early polymers from dimers up to hexamers. In the present work the latter type alone are studied and many alternative mechanisms are suggested, of which, owing to the complexity of structure involved, no one can be considered more satisfactory than another. M. McL.

1403. Dimerization of Petroleum Hydrocarbons. W. J. Sparks, R. Rosen, and P. K. Frolich. *Trans. Faraday Soc.*, 1939, **35**, 1040–1054.—Starting from a description of the commercial dimerization of *iso*butene to obtain commercial *iso*-octane, the authors develop a theory of dimerization involving the shift from one monomer to another of a hydrogen atom which finally appears in the dimer on the previously unsaturated C-atom which adjoins the newly formed bond. This shift may occur through free valencies or through a prototropic change.

Other dimerization reactions are discussed, notably those of olefines, di-olefines, and acetylene, and it is shown that the proposed shift is, in general, not at variance with the apparent mechanisms involved. M. McL.

1404. Preparation and some Physical Properties of 2 : 2 : 4 : 4-Tetramethylpentane. F. L. Howard. *Bur. Stand. J. Res.* (*Wash.*), 1940, **24** (6), 677–684.—The method of Whitmore and Southgate was used, and consists in reacting dimethyl zinc with 2 : 2 : 4-trimethyl-4-chloropentane. The latter was prepared by the action of HCl on "di*iso*butylene," whilst the zinc dimethyl was made by reacting methyl iodide with zinc copper alloy.

The crude 2 : 2 : 4 : 4-tetramethylpentane was purified and then distilled. The physical constants determined on the purest fraction included freezing point, boiling point, and its variation with pressure, refractive index, and density, and their variations with temperature. D. L. S.

Analysis and Testing.

1405.* Determination of Naphthalene in Coal Tar and its Products. W. Kirby. *J. Soc. chem. Ind.*, 1940, **59**, 168–171.—The method is based on the fact that the addition of tar oil to naphthalene lowers the setting point by an amount depending on the quantity of oil added.

The determination of naphthalene in coal-tar naphtha is given as an example :—

The residue left on distilling 100 gm. of naphtha to 197° C. was 7·5 gm. A portion of this residue weighing 4·992 gm. was added to 14·630 gm. of pure naphthalene. The mixture was melted, and its setting point was found to be 70·8° C., corresponding with 83% of naphthalene. A simple calculation shows that the naphthalene originally present in the naphtha was 2·4%. Normally it is preferable to stop the distillation for a naphthalene residue at 190° C.

Curves and tables are presented giving the percentage naphthalene in coal-tar products according to setting point, in quantities ranging from 30% to 100% naphthalene. T. C. G. T.

1406.* Determination of the Composition of Commercial *iso*Pentane. K. H. Hachmuth and J. W. Tooke. *Nat. Petrol. News*, 1.5.40, **32** (18), R. 142.—The anti-knock characteristics of commercial *iso*pentane are related to the composition. Low concentrations of *n*-pentane and butane which reduce the octane number of the commercial product quantitatively may be determined accurately by the procedure outlined. The method employs a modification of the standard A.S.T.M. distillation apparatus and is based on a precise determination of the boiling temperature curve. The accuracy claimed is 0·5% for *n*- and *iso*pentane and 0·25% for *n*-butane.

H. G.

1407. Determination of Total Water-Soluble Chlorides in Petroleum. J. W. Horne and L. F. Christianson. *U.S. Bur. Mines Report of Investigations*, No. 3517, June 1940.—In order quantitatively to determine the amount of salt in petroleum, it is necessary first to extract the salt from the oil, and secondly to measure the amount of extracted salt. To be effective the procedure adopted must be universally applicable to the various emulsions and salts which accompany petroleum; it must be accurate and precise; and finally it must be adaptable to either field or laboratory analysis. The technique used by the Bureau of Mines and described in this paper is claimed to fulfil all these conditions.

A representative sample of the oil is obtained and thoroughly mixed, after which a portion is measured for analysis. The measured oil is then diluted with xylene to dissolve wax, gum, and asphalt. To prevent any emulsion being formed, or to resolve any that has been formed, a stabilizer is added and the mixture heated and agitated violently to dissolve the salt. The oil is then separated from the water by centrifuging and the total quantity of water extract titrated for the dissolved chlorides. The quantity of water-soluble chlorides present in the oil is calculated as sodium chloride from the net millilitres of silver nitrate used in the titration. H. B. M.

1408.* Some Notes on Exhaust Gas Analysis. H. Fossett. *J. Instn aut. Engrs*, 1940, VIII (9), ix–xxiv.—The determination of exhaust-gas composition has a considerable field of usefulness in the testing and control of internal-combustion engines. The author first reviews published work on the composition of exhaust gases, and then outlines various methods of analysis.

Ballantyne's empirical rule for the relationship of H_2 and CH_4 to CO content has long been established, but was disproved in 1936 by D'Alleva and Lovell, who carried out very careful experiments on three engines. Data for the relationship of CO_2, CO, and O_2 to air/fuel ratio are shown graphically. It was found that the CH_4 produced was constant, being about 1·857% of the carbon in the fuel; this is equivalent to the formation of 0·15 mols. of CH_4 per molecule of C_8H_{17}, which was the composition of the fuel used.

Methods of exhaust gas analysis are: (*a*) Chemical analysis, (*b*) hot-wire catalysis, (*c*) relative density and (*d*) thermal conductivity. For chemical analysis the Orsatt apparatus is extremely useful. The thermal conductivity principle is most widely used for portable instruments recording air/fuel ratio. Differences exist in the calibration of different designs of these instruments; it would appear that a linear scale is incorrect. A characteristic of conductivity instruments is that, if the mixture strength is progressively weakened, beyond 14·7 : 1 the pointer will move back towards the rich end of the scale. Considerable caution must therefore be exercised when operating at the weak end of the scale. K. A.

Motor Fuels.

1409. Co-operative Fuel Research Motor-Gasoline Survey, Winter 1939–40. E. C. Lane. *U.S. Bur. Mines Report of Investigations*, No. 3524, June 1940.—The Co-operative Fuel Research Committee and the Bureau of Mines have already issued eight reports on properties of commercial motor fuels. This, the ninth, embodies data on 2033 samples, representing the products of eighty different companies. In addition, there are included data compiled by the Pacific Coast Technical Group for that region. For the purposes of this report, gasolines are again divided into regular-price, premium-price, and third-grade qualities. Table 1 gives data on gravity, sulphur content, vapour pressure, octane number, and distillation characteristics of commercial gasolines sold in twenty-one marketing areas in the U.S. during 1939–40. Averages for each of the grades are included in this table. These averages are assembled in Tables 2, 3 and 4. Table 5 records the minimum and maximum for each characteristic of the three grades of gasoline. Table 6 is for comparative purposes, and includes the same figures for the previous winter survey. H. B. M.

1410. Volatility and Anti-knock Properties of Hydrogenated Gasoline. A. V. Agafonov and C. K. Makarov. *Neft. Khoz.*, 1939, No. 8, 40.—Volatility characteristics and octane numbers have been determined for a number of Russian aviation gasolines, and various blends with *iso*-octane, light gasoline fractions, and alkylbenzene, with and without $PbEt_4$.

A method is given for the treatment of pressure distillate by means of which a high octane blending agent of high volatility is obtained and by reforming the residue of the pressure distillate a high-grade aviation petrol is produced. Blending hydrogenated light fractions of pressure distillate with i.b.p. of 60° C. for obtaining 100 octane fuel reduces the consumption of *iso*-octane 25–40%. The following composition is recommended: 15% light fractions + 30% *iso*-octane + 55% aviation gasoline. In this blend either straight-run gasoline may be used or hydrogenated gasoline obtained by reforming cracked ligroine. The distillation characteristics and the vapour pressure of this 100 octane fuel are within the specifications for aviation gasoline.

It is also possible to obtain 100 octane fuels if in the blend *iso*-octane is replaced by the fraction distilling between 120° and 165° C. of alkylbenzene, which is not obtained by blending alkylbenzene with gasoline alone. L. R.

1411. Patents on Motor Fuel. D. Lipkin. U.S.P. 2,210,942, 13.8.40. Appl. 20.10.36. Preparation of a fuel for internal-combustion engines by admixing a hydrocarbon of the motor fuel boiling range with a quantity of *iso*butyraldehyde to increase substantially the anti-knock value.

F. R. Bean. U.S.P. 2,211,313, 13.8.40. Appl. 1.7.38. Preparation of a motor fuel consisting of hydrocarbons which normally tend to deteriorate and a lower monoalkyl ester of *p*-hydroxyphenylamino acetic acid in an amount sufficient to inhibit said deterioration.

A. N. Sachanen and A. W. Francis. U.S.P. 2,211,732, 13.8.40. Appl. 10.2.39. Preparation of a motor fuel having the distillation characteristics of an aviation gasoline and a smooth boiling range. The fuel consists of a mixture of alkylbenzenes boiling above 220° F. and a gasoline fraction boiling below about 220° F.

Standard Oil Development Corp. E.P. 524,252, 1.8.40. Appl. 26.1.39. Preparation of hydrocarbons boiling mainly within the gasoline range by contacting with an alkylation catalyst a mixture of non-cyclic paraffin hydrocarbons containing a paraffin hydrocarbon having at least one tertiary carbon atom in the molecule and a further paraffinic hydrocarbon. H. B. M.

Lubricants and Lubrication.

1412. Theoretical and Experimental Researches on the Synthesis of Lubricants. M. G. Hugel. *Trans. Faraday Soc.*, 1939, 35, 1054–1061.—The above aspects of the synthesis of lubricating oils from rubber are here discussed. The author applies Dunstan's and Thole's molecular weight/viscosity law to Andrade's viscosity/temperature equation to discover what chemical constituents are responsible for varying viscosity effects. The differences between mineral and vegetable oils he considers due possibly to a dipole effect in the molecules of the latter.

In the synthesis of oils from rubber by hydrogenation under high pressure three reactions occur: cracking, cyclization, and hydrogenation. Catalysts can be chosen so as to determine which of these three effects is predominant. The author considers aliphatic oils best for lubrication below 300° C., mixed type for oils required to volatilize, and polycyclic aromatics for very high temperatures. M. McL.

1413. Regeneration of (Used) Oils. E. Schmitz. *Science et Ind.*, 1940, 290 bis. (Supplement to *Revue Mécanique*), 77–83.—The author puts the case for a national scheme for the recovery of used oils, particularly in war-time, pointing out that the practice is obligatory in Germany. The methods of regeneration are discussed together with the quality of oils so recovered. It is suggested that for efficiency and economy the oils be collected and regenerated in six different classes, viz.: (1) Light machinery, (2) Electrical and Turbine, (3) Automobile, (4) Aviation, (5) Marine diesel engine, (6) Rough industrial machinery oils, each group to be treated by the method appropriate to the quality required by the finished product. R. D. S.

1414. Use of Bleaching Earths in the Regeneration of Lubricating Oils. F. Bloemen. *Science et Ind.*, 1940, 290 bis. (Supplement to *Revue Mécanique*), 84–85.—The action of an earth may depend on absorption by the pores of the earth or adsorption by the activated surface, or in many cases chemical action by the particles. The latter appears to be due to the acid nature of the earth and to the tendency of the particles to behave as ions in consequence of the acceptance of an electric charge. Whether a particular earth is used or not depends on the oil to which it is to be applied, for the order of merit of a series of earths is not the same for different oils, since one of the above-mentioned mechanisms will be more effective on one oil than on another. Hence the choice of earth depends on the type of oil, and the type of refining to be employed. This is sometimes difficult, for often the oil to be treated is a mixture of, *e.g.*, diesel and aviation oil; especially is this so if one of the components is compounded. It is therefore suggested that there should be either a central laboratory for the analysis of used oils for recovery or, alternatively, each type of oil should be collected separately. R. D. S.

1415.* Deterioration Factors in Diesel Lubricants. C. G. A. Rosen. *Nat. Petrol. News*, 1.5.40, 32 (18), R.152.—The factors causing the deterioration of diesel lubricant include the time that the oil is in effective use and the frequency with which it is subject to heat, oxidation, or contaminating influences. Used oil, in some respects, is better than new oil. It has been found that used oil, particularly if compounded, has a higher film strength and often higher resistance to cylinder corrosion than new oil. On the other hand, new oil is less likely to cause bearing corrosion. The most important factor governing oil life is temperature, and all efforts should be made to reduce crankcase temperature. Flame impingement on cylinder walls causes local hot spots and aggravates cylinder wall deposits. Bearings should be designed with rapid heat dissipation in view. A thermal sponge behind a thin layer of babbitt has been found satisfactory. Since the diesel engine is an oxidizing apparatus, lubricants should show a maximum resistance to change due to oxidizing influences. H. G.

1416.* Synthetic Fats in Grease-making. A. W. Burwell and J. A. Camelford. *Petroleum*, 1940, 2 (3), 75–78.—One of the most ready sources of supply of saponifiable materials which can be used for grease manufacture is the oxidation under controlled conditions of petroleum hydrocarbons. Among the products formed in this process quantitative yields are obtained of acids, primary and secondary alcohols, esters, keto-alcohols, lactones, and ketones. Many of these oxidation products are readily saponifiable with bases ordinarily used in grease manufacture, and soaps of Na, Ca, Al, Pb, K, Mg, and Ba have been prepared by direct saponification and used in lubricating greases.

It is claimed that the greases so obtained are free from bleeding in storage, from separation after working, and that most of them after heating above their melting point return to their original consistency without separation.

The method of manufacturing the greases is outlined. It is essential that these products be finished exactly neutral or slightly acidic, never alkaline. Barium greases have been made very satisfactorily by direct saponification with $Ba(OH)_2$ and showed worked penetrations of 230–280 at 77° F. and melting points above 350° F. In addition, such products could be heated to 420° F., and on cooling were identical with the original grease.

The soaps can also be made by double decomposition and then dissolved in the appropriate mineral oil base.

Experiments were carried out in the manufacture of aluminium stearate greases. It was found that if this soap were blended with mineral oil containing a small amount of some of the esters, or mixtures of esters with high-molecular-weight ketones and alcohols prepared from petroleum hydrocarbons, a greatly improved grease resulted.
D. L. S.

1417. **Patents on Lubricants and Lubrication.** W. H. James. U.S.P. 2,209,976, 6.8.40. Appl. 20.1.38. Preparation of a mineral-oil composition consisting of a refined viscous mineral oil and a small amount of an N, N^1-dithiobisalkylamine.

I. M. Colbeth. U.S.P. 2,210,140, 6.8.40. Appl. 6.11.31. Lubricant consisting of a major proportion of mineral oil and a minor proportion of an ester derived from a long-chain aliphatic acid and a short-chain aliphatic alcohol having not more than two hydroxyl groups. The amount of the ester is at least 0·5% of the lubricant.

W. A. Whittier, N. D. Williams, and H. L. Moir. U.S.P. 2,211,306, 13.8.40. Appl. 19.12.35. Preparation of a lubricant by chemically combining a small amount of sulphur with a fatty body, adding phosphorus to the sulphurized body, and heating to a temperature not much in excess of 230° F. until the reagents have prevented substantial fuming of phosphorus.

B. Folda. U.S.P. 2,211,373, 13.8.40. Appl. 8.8.39. Preparation of a thread lubricant consisting of a cup grease and zinc dust rendered chemically stable by the addition of a free fatty substance in amount sufficient to neutralize the free alkaline ingredients of the cup grease and to leave an excess of at least 1/10 of 1% by weight of the finished compound.

E. M. Steffen. U.S.P. 2,211,442, 13.8.40. Appl. 15.9.36. Preparation of synthetic compositions useful for blending with lubricating oils. Rubber material is subjected to the action of anhydrous aluminium chloride in the presence of metallic iron under conditions effective to produce conversion products.

B. T. Brooks. U.S.P. 2,211,687, 13.8.40. Appl. 18.3.37. Preparation of a lubricant consisting of a mineral lubricant and a small amount of stearone.

Carbide and Carbon Chemicals Corp. E.P. 524,847, 15.8.40. Appl. 18.2.38. Production of a water-dispersible composition for use in textile lubricants comprising a major proportion of an oil, and a lesser quantity of a surface active agent consisting either of a 1, 2 di-substituted glyoxalidine or of a carboxylic acid addition product of these compounds.
H. B. M.

See also Abstract No. 1419.

Gas, Diesel and Fuel Oils.

1418.* **Diesel Fuel in Houdry Distillate Close to Straight-run Quality.** W. S. Mount and E. T. Scafe. *Nat. Petrol. News*, 10.7.40, **32** (28), R. 246.—When the limit of the availability of straight-run diesel fuel of suitable quality is reached, the adaptability of the Houdry catalytic cracking process is important. A typical cetane number of fuel so produced is 46, as compared with 35 for thermal cracked fuel. Catalytically cracked fuels from Pennsylvania and Oklahoma crudes have only slightly lower cetane numbers than straight-run products of similar characteristics, and they yield equally well to ignition accelerators. In other respects they resemble the straight-run distillates.
H. G.

1419.* Diesel Fuel and Lubricants. L. C. Welch. *Nat. Petrol. News*, 15.5.40, 32 (18), 38.—There is a growing tendency to use the light grades of domestic heating oils for diesel fuel, and also to model specifications on that of light fuel oil. Significant factors in present-day specifications are: Ignition quality (cetane number), cleanliness, viscosity (to minimize pump wear), pour point, distillation range (to provide clean combustion), sulphur, alkali and acid contents (to minimize corrosion). Opinions relative to the type of lubricant best suited to the diesel engine differ. The present tendency is to emphasize the need for low carbon forming and oxidation tendencies. The use of inhibitors is growing, and in some cases detergents are added in order to increase the scavenging of deposits. H. G.

Asphalt, Bitumen and Road Construction.

1420. Subgrade Pressures under Flexible Road Surfaces. A. T. Goldbeck. *Highway Research Board Proc. Nineteenth Annual Meeting*, 1939, 164–174.—The author presents results of preliminary tests made to determine the subgrade pressures under flexible road surfaces of varying thickness. The tests were made on soil–clay mixtures, containing sufficient water to give the requisite stability, contained in a wooden bin.

The mixture was rolled in 3-in. layers with a sheep's foot roller, followed by a heavy hand concrete roller.

The surfacing layer was composed of stone screenings of different thickness.

Loads were applied through elliptical bearing blocks having areas equal to the area of contact of tyres suitable for loads of 4000, 8000, and 12,000 lb., and soil pressure measurements were made by means of the soil pressure cell described in a previous paper.

Load-indentation tests were also made in order to determine whether the maximum subgrade pressures were greater or less than the subgrade supporting value.

The apparatus consisted of steel bearing blocks through which the load was applied in increments by means of a hydraulic jack, the indentation being measured by means of two Ames dials reading to 0·001 in. In making a test a hole was formed in the surfacing material and a flat level surface then made in the subgrade material to support the bearing block. Tests on the surfacing material were also made in a similar manner. A. O.

1421. Present Design Practice and Construction Developments in Flexible Pavements. B. E. Gray. *Highway Research Board Proc. Nineteenth Annual Meeting*, 1939, 175–192.—The author summarizes the replies to a questionnaire concerning the practice of various State highway departments in design and construction of bituminous surfacing. A. O.

1422. Report of Committee on Fillers and Cushion Courses for Brick and Block Pavements. J. S. Crandell. *Highway Research Board Proc. Nineteenth Annual Meeting*, 1939, 275–278.—Details of construction and results of tests on the Brick Filler Test Road in Ohio were given in previous reports, and the present report summarizes results of investigation of the changes in physical characteristics and conditions of five asphaltic bitumen fillers during four years' service.

Observations were made on the pavement surface condition; small areas of the bricks were removed, the adhesion of filler to brick was noted, and samples of filler prepared for testing by cleaning and melting. Results of tests on the recovered filler are compared with tests on the original fillers. All fillers showed an appreciable increase in softening point, decrease in ductility and penetration, and increase in asphaltene content. No consistent relationship was observed between the change in properties and type of petroleum base. Exudation of the filler appears to be greater with fillers of lower softening point and higher penetration at 46° C. With the fillers employed, bonding of filler to brick and low exuding properties appeared to be incompatible, but no loose bricks were observed in any section, thus indicating that the fillers behave satisfactorily without bonding to the brick. A. O.

1423. Status of Soil Stabilization in United States, December 1939. W. H. Mills. *Highway Research Board Proc. Nineteenth Annual Meeting*, 1939, 491–497.—The author summarizes the replies to a questionnaire sent to State highway departments requesting a report of the mileage of all types of stabilized road constructed since 1924.
A. O.

1424. Soil Stabilization with Emulsified Asphalt in the Mid-Continent Area. B. S. Scoggin. *Highway Research Board Proc. Nineteenth Annual Meeting*, 1939, 498–503. —Details are given of emulsified asphalt soil stabilization projects covering an area equal to 225–250 miles of 18-ft. roadway. Cost of the work ranged from 22 to 28 cents per yard of 4 in. compacted thickness, and the following conclusions were reached from the work : maximum dehydration is a more important factor affecting durability than maximum density at time of compaction. The quantity of emulsion should be determined by test mixtures of the emulsion and representative samples of the soil using some known efficiency test such as the McKesson. Four-inch compacted depth should be the minimum thickness, while if the base is a subsoil of high plastic index, the minimum should be 6 in. Control tests should be made on the finished work to ensure that the material laid meets the minimum requirements of the efficiency test.
A. O.

Special Products.

1425. Fatty Acids from Paraffin Wax—Purification for Soap Making Purposes. Anon. *Chem. Tr. J.*, 19.7.40, 107, 38.—Fatty acids obtained by the catalytic oxidation of paraffin waxes (*e.g.*, those from Fischer–Tropsch plants) contain, in addition to the straight acids, oxy-acids and internal lactones which cannot be completely removed by saponification, followed by extraction or distillation, and recovering the fatty acids by acidification. The resulting acids are liable to discolour and have an unpleasant odour. A process has now been worked out by the I.G. Farbenind. A.-G. (E.P. 518,963 of 13th March, 1940) for the purification of the crude fatty acids by fractional distillation below 10 mm. pressure of mercury (preferably at 2 mm.), removing the fatty acids with acid value >330 at a temperature up to about 130° C., and then recovering with the application of steam under 2 mm. mercury, the soap forming fatty acids, boiling up to 260° C. with acid value 200–330. Complete separation of the fatty acids consists of saponification of the oxidation products, removal of unsaponifiable components by extraction with aqueous ethyl alcohol, followed by benzine and decomposition of the soaps with 50% of H_2SO_4. The crude fatty acids are finally purified by vacuum fractional distillation as described in the patent.
C. L. G.

1426. Patents on Special Products. G. W. Johnson. E.P. 523,892, 25.7.40. Appl. 9.9.38. Manufacture of meta-diaza-compounds by heating cyclic compounds with ammonia and aldehydes or compounds splitting off aldehydes.

A. G. Green. E.P. 524,011, 29.7.40. Appl. 25.10.38. Manufacture of a product having valuable therapeutic properties by the reaction of an acetic aldehyde bisulphite on para-aminobenzene-sulphonamide.

Consortium für Elektrochemische Industrie, G.m.b.H. E.P. 524,086, 30.7.40. Appl. 21.1.39. Manufacture of hydroxycarboxylic acid esters by the catalytic reduction of formic ester condensation products with carboxylic esters.

Röhm and Haas Company. E.P. 524,151, 31.7.40. Appl. 23.1.39. Preparation of *iso*phorones containing at least 15 carbon atoms by heating a saturated alkyl monoketone with a member of the group consisting of an oxide, hydroxide, amide, and alkoxide of an alkali metal at a temperature above 100° C., so as to split out water.

The Distillers Company, Ltd. E.P. 524,156, 31.7.40. Appl. 23.1.38. Production of polymers of styrene by subjecting a solution thereof to the action of an activating bleach earth, an activated clay and/or silica gel at a temperature of 20–90° C.

E.I. Du Pont de Nemours. E.P. 544,163, 31.7.40. Appl. 23.1.39. Manufacture of saturated aliphatic carboxylic acids by oxidizing long-chain unsaturated fatty acids with highly concentrated nitric acid at ordinary or slightly elevated temperature in the presence of a vanadium catalyst.

N.V. Orgachemie. E.P. 524,319, 2.8.40. Appl. 27.1.39. Preparation of stable concentrated quinine solutions by dissolving a mono-salt of quinine with the aid of ascorbic acid.

I.G. Farbenindustrie A.-G. E.P. 524,349, 5.8.40. Appl. 27.1.39. Production of melamine by acting with gaseous anhydrous ammonia at elevated pressure on dicyandiamide at a temperature above 100° C.

E.I. Du Pont de Nemours. E.P. 524,470, 7.8.40. Appl. 30.1.39. Manufacture of alkoxy-*cyclo*hexanols by reacting *cyclo*hexene oxide with a monohydric aliphatic alcohol of at least 6 carbon atoms.

J. W. C. Crawford. E.P. 524,521, 8.8.40. Appl. 31.1.39. Manufacture of organic condensation products by condensing an olefinic compound derived from a chlorinated paraffin hydrocarbon with acids or acid anhydrides of specific formula.

I.G. Farbenindustrie A.-G. E.P. 524,696, 13.8.40. Appl. 3.2.38. Manufacture of *para*-aminobenzaldehydes by reacting a secondary N-alkylaniline with a phosphorus oxychloride and formyl-N-methylaniline.

H. E. Potts. E.P. 524,715, 13.8.40. Appl. 4.2.39. Improvement in a process for the treatment of liquid distillable carbonaceous materials containing solid matter in dispersion with hydrogen or gases containing free hydrogen. A tube preheater provided with return bends is employed, and interchangeable tube-like pieces constructed of brittle ceramic materials or the like are inserted in the return bends.

Carbide and Carbon Chemical Corp. E.P. 524,759, 14.8.40. Appl. 15.3.38. Manufacture of olefine oxides by the direct chemical combination of molecular oxygen with olefins. A gaseous mixture containing oxygen and olefine is passed through a confined space containing a catalyst at a temperature between 150° and 400° C. The amount of olefine oxide produced per unit volume of catalyst present is controlled by maintaining a mass velocity of the gaseous mixture of not less than 0·2 lb. per sq. ft. per second.

H. E. Potts. E.P. 524,760, 14.8.40. Appl. 7.2.39. Use of mixtures of metal sulphides as catalysts in the production of hydrocarbons from carbonaceous materials. The catalysts are obtained by mixing in a dry state ammonium thio salts of metals of the sixth group of the periodic system with carbonates or salts of organic acids of metals of the iron group and heating the mixture at temperatures between 300° and 500° C. at least until the formation of ammonia ceases.

Consortium für Elektrochemische Industrie, G.m.b.H. E.P. 524,849, 15.8.40. Appl. 9.2.39. Manufacture of butadiene from 1 : 3-butylene glycol by splitting off water in the presence of an amount by volume of water many times as great as the amount of butylene glycol present.

G. W. Johnson. E.P. 524,918, 19.8.40. Appl. 13.6.38. Production of butadiene by dehydrogenation of butylene. A gaseous mixture of normal butylene is led with steam at a temperature between 500° and 700° C. over a catalyst consisting of more than 50% zinc oxide, together with one or more oxides or salts of the oxy-acids of chromium, vanadium, molybdenum, uranium, or tungsten.

I.G. Farbenindustrie A.-G. E.P. 525,136, 22.8.40. Appl. 14.2.39. Manufacture of mustard oils by heating chlor-olefines in which a chlorine atom is situated in α-position to an olefinic double bond with an aqueous solution of thiocyanic acid salt.

Carbide and Chemicals Corp. E.P. 525,142, 22.8.40. Appl. 15.2.39. Preparation of *iso*phorone by contacting acetone vapours with a condensation catalyst at a temperature between 200° and 700° C.

Vereinigte Glanzstoff-Fabriken A.-G. E.P. 525,147, 22.8.40. Appl. 15.2.39. Preparation of amines of petroleum hydrocarbons. Raw or purified mineral oil is treated with chlorine whilst cooling, and ammonia is caused to act on the chlorinated product in the presence of indifferent solvents such as methyl alcohol, ethyl alcohol, which are capable of dissolving simultaneously ammonia and chlorinated petroleum to a considerable extent.

Wingfoot Corp. E.P. 525,172, 22.8.40. Appl. 16.2.39. Preservation of rubber or oils by treating them with the reaction product of diethylene glycol or monoalkyl ether thereof with an amine in which each amino-group present has at least two aromatic substituents on the nitrogen.

A. L. Stillman. E.P. 524,991, 20.8.40. Appl. 12.1.39. Manufacture of a stable liquid fuel having coal particles equally distributed throughout and consisting of a mixture of 40–70% by weight of fuel oil and from 60–30% of coal of the bituminous type which has been subjected to a grinding action in the presence of oil so that it passes a 325-mesh sieve. H. B. M.

Detonation and Engines.

1427. Diesel Engines Underground. I. Composition of Exhaust Gas from Engines in Proper Mechanical Condition. *Report of U.S. Dept. of the Interior,* May 1940. J. C. Holtz, L. B. Berger, M. A. Elliott, and H. H. Schrenk.—An investigation of the suitability of diesel engines for underground use in mines has been initiated by the U.S. Bureau of Mines. This report—the first of a series—deals with the composition of exhaust gases over the rated operating ranges of two commercial four-stroke diesel engines, excluding the effect of such factors as excessive wear, maladjustment, etc. The effect of exceeding the maximum fuel quantity allowed by the maker's adjustment was also studied.

The engines were four-cylinder units, rated at 44 and 70 max. b.h.p., respectively. A fuel of 78 cetane number was used. Exhaust gas samples were collected for determination of CO_2, O_2, CO, CH_4, H_2, and N_2. The exhaust pipe was extended about 9 ft. beyond the sampling points; this proved necessary to avoid contamination with air drawn back into the pipe by pulsations. The methods of sampling and analysis are described in detail, together with the calculations and a discussion of probable accuracy.

Graphs are presented correlating exhaust-gas composition with fuel–air ratio as well as air–fuel ratio. It is pointed out that the former has advantages when dealing with the wide range of ratios occurring in diesel operation. The relation between composition and fuel–air ratio was the same for both engines. As long as the rated maximum load was not exceeded the CO content was sufficiently low to permit adequate dilution and safe removal from underground workings. If this load was exceeded, the CO hazard was equal to that of the gasoline engine. The CO concentration was a minimum at 0·03 : 1 fuel–air ratio; at still leaner ratios it increased somewhat.

The concentration of oxides of nitrogen was also a maximum at about 0·03 : 1 fuel–air ratio, when values of 440 and 676 parts per million by volume were observed. This is dangerous for even short exposures; thus, oxides of nitrogen constitute a hazard to be considered.

Aldehyde concentration reached 5 p.p.m. in one engine and 31 p.p.m. in the other, the maximum occurring at low fuel–air ratios. It is suggested that exhaust odour and irritating effect may be related to aldehyde concentration, but the data are not complete. The concentration of CO_2 was between 2% and 13%; this gas must also be considered when estimating ventilation requirements. Soot appeared to be present in the exhaust even when excess air was available.

The chemical aspects of the diesel combustion process are discussed in relation to the published work of Boerlage and Broeze. These investigators postulated that combustion is characterized by two simultaneous processes: direct oxidation and thermal decomposition. It is concluded that the above results confirm this idea.

K. A.

1428.* Octane Number Problem from the Automotive Viewpoint. J. B. Macauley. *Nat. Petrol. News*, 1.5.40, **32** (18), 30.—An examination of figures relating to average compression ratios and average octane numbers over the past 10 years shows that each function has increased in a similar manner. It is reasonable to suppose, therefore, that if octane numbers continue to increase, engine design will progress in order to profit by the increase in potential power. The use of superchargers is suggested as a feasible means of attaining greater power outputs. Engine factors militating against extra power production are: Variations in spark setting, combustion-chamber deposits, variations due to manufacturing tolerances, variations in fuel type and type of operation. The quantitative effect of these factors is discussed. The author suggests the routine application of solvent processes to minimize combustion-chamber deposits. H. G.

1429.* New Fuel Data from Tests Show How Maintenance Improves Mileage. Anon. *Nat. Petrol. News*, 22.5.40, **32** (21), 36.—In a series of road tests a representative number of 1940 passenger cars were driven for 100,000 miles at an average speed of 50 m.p.h. Full details are not yet available, but the results so far indicate that the average top speed after 100,000 miles was only 2 m.p.h. lower than that of a second group of cars driven only 1700 miles. The average time taken to accelerate from 10 to 50 m.p.h. showed practically no change, and gasoline mileage per gallon showed a slight rise at the end of the tests. H. G.

Economics and Statistics.

1430. Natural-Gasoline Plants in the United States, 1st January, 1940. G. R. Hopkins and E. M. Seeley. *U.S. Bur. Mines Information Circular*, No. 7126, July 1940.—Figures included in the Bureau of Mines' latest biennial survey of natural-gasoline plants in the U.S. reveal that the tendency noted during the past 10 years for manufacture to be concentrated in fewer establishments has now been checked. On 1st January, 1940, there were 729 plants in existence, or only twelve fewer than on 1st January, 1938; 671 of these were in operation and the remainder shut down. The total daily capacity of these plants was the highest ever recorded, and showed an increase over the January 1938 figure of 5%. Owing to a hold-over of capacity built before proration, and equally to the continued practice of building up to peak gas production, a large percentage of this capacity was idle. In 1938 and 1939 it is estimated that idle capacity was in the region of 47% and in 1937, 40%.

Texas, California, and Oklahoma together accounted for 84% of total operating capacity on both 1st January, 1938, and 1st January, 1940. The appearance of recycling plants designed to process the gas for condensate and return the residue gas to producing formations was reflected in this survey. The returns included ten such plants, each with a capacity of 246,000 gal. H. B. M.

1431.* World Production of Petroleum—Venezuela. J. Bermejo. *Bol. Inform. Petroleras*, April 1940, XVII (188), 82–92.—Venezuela commenced production in 1917 with some 18,000 tons, rising to the important figure of 30½ million tons in 1939. The article quoted gives interesting particulars, both historical and statistical, of the remarkable growth of the oil industry. A full bibliography is added. H. I. L.

BOOK REVIEW.

Hydrocarbon Chemistry—A General Discussion reprinted from the Transactions of the Faraday Society. Pp. 287. Gurney and Jackson, 98 Gt. Russell Street, London. Price 12*s.* 6*d.*

This publication covers the papers presented at a three-day Discussion on Hydrocarbon Chemistry held by the Faraday Society.

The volume commences with an introductory paper contributed by the President, Professor E. K. Rideal. The subject-matter is then considered under the following heads :—

Part I.—Homogeneous Thermal Reactions of Hydrocarbons.
Part II.—Catalytic Reactions of Hydrocarbons.
Part III.—The Mechanism of the Technical Synthesis and Transformation of Hydrocarbons.
Part IV.—Olefine Polymerization.

Professor Rideal explains in his introductory paper that while the technical development of certain phases of hydrocarbon chemistry has attained a high standard, the theoretical aspect is confused in its general outlook. The main official purpose of the Discussion, therefore, has been to direct the attention of those academically inclined to the wide variety of such problems which await solution and elucidation, and in consequence the book is academic rather than technical in character.

The first three papers in Part I deal mainly with structure and activation energies of hydrocarbon molecules, the remaining nine papers in this part giving an account of certain of the views held on homogeneous thermal reactions of hydrocarbons. This section is of particular importance to the petroleum technologist, for it deals with many matters which are the basis of the thermal cracking process, covering a wide field from chain reactions to the pyrolysis of hydrocarbon groups and individual hydrocarbons.

Part II is devoted to catalytic reactions of hydrocarbons. A comprehensive paper on catalytic ring closure of open-chain hydrocarbons, by Professor Taylor and Dr. Turkevitch, and two papers by Dr. Farkas on isomerization occupy most of this interesting section.

In Part III the technical synthesis and transformation of hydrocarbons is considered. In the first two papers the mechanism by which long-chain hydrocarbons are built up from water-gas in the Fischer–Tropsch process is discussed. The third paper, contributed by Dr. Pier of the I.G. Farbenindustrie, deals with the coal-hydrogenation process. The important subject of cyclization of saturated straight-chain to aromatic hydrocarbons has attracted three papers. Divergent views as to the mechanism of the reaction are put forward by Mr. Pitkethly and Dr. Steiner of the Sunbury Research Station, and by Dr. Hoog and his collaborators of the Bataafshe Petroleum Maatschappij. The recently discovered and most interesting reaction in hydrocarbon chemistry—namely, the catalytic condensation of an olefine to an *iso*-paraffin—is excellently dealt with by Drs. Dunstan and Birch. These authors are to be congratulated on their sound and careful work on this important and elegant method, which is destined to have profound and far-reaching effects on hydrocarbon technology.

The last part, Part IV, is devoted to olefine polymerization, in which papers embracing the formation of dimeric molecules, of medium-length molecules for use as lubricants, and of high-molecular-weight plastics are presented.

This book is undoubtedly one of the best publications on hydrocarbon chemistry and comprehensive surveys from a research standpoint which has appeared in

English. At the published price of 12*s*. 6*d*. it is a noteworthy bargain. Many of the views put forward are controversial, and divergent opinions are the rule rather than the exception. This is one of the most important functions of the work, in so far as it has collected and recorded a consensus of opinion of admitted authorities on hydrocarbon chemistry. The volume is so definitely academic that it will make little appeal to the technologist, but will be extremely valuable to the chemist and research worker.

T. G. HUNTER.

BOOKS RECEIVED.

Petroleum Development and Technology, 1940. Trans. of the American Institute of Mining and Metallurgical Engineers, Vol. 136. Pp. 608. Published by the Institute, 29 West 39th Street, New York, U.S.A. Price $5.

This volume is the fifteenth of the Petroleum Development and Technology Series of the Petroleum Division A.I.M.E., and contains papers presented before the Division during the year under review. Abstracts of the most important papers have already appeared in the abstracts section of the *Journal of the Institute of Petroleum.*

There are five chapters, devoted respectively to (1) Production Engineering, (2) Engineering Research ,(3) Petroleum Economics, (4) Production, Domestic (U.S.A.) and Foreign, and (5) Refining.

British Standard Methods for the Sampling and Examination of Bituminous Road Mixtures. (Revised August, 1940.) No. 598—1940. Pp. 41. British Standards Institution, 28, Victoria Street, London, S.W. 1. Price 3*s*. 6*d*.

Experience gained in the adoption of the first issue of this British Standard (published in February 1936) showed that a number of modifications were desirable. The following are some of the more important modifications which have been incorporated in this present revision :—

(*a*) The size of the samples required for testing has in nearly all cases been increased.

(*b*) The test for the determination of bitumen content has been revised.

(*c*) A modified test for the rapid determination of bitumen content which gives a good approximation is outlined in an appendix as being useful in those laboratories where routine testing is carried out.

(*d*) The two alternative methods for the recovery of bitumen which have been included give similar results.

(*e*) The tests on mineral aggregate have not been included, as they are provided for in B.S.S. 812.

(*f*) The section dealing with the identification of mineral aggregate has been completely revised.

INSTITUTE NOTES.

October 1940.

MEMBERS SERVING WITH H.M. FORCES.

The following is the first list of members of the Institute who are serving with H.M. Forces. The information is correct to the date of notification, though incomplete. Promotions and transfers may have been made subsequently. The Secretary will be pleased to receive additional information from time to time both as regards members in this list and members from whom information has not yet been received. Such additional information will be incorporated in a subsequent list.

Adlington, R. E., Capt., R.A.S.C.
Bartley, E. B., Cadet.
Blake-Smith, C., Flight-Lieut., R.A.F.
Boshell, E. G., R.A.F.
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AWARD OF THE GEORGE MEDAL.

The Council warmly congratulates Mr. **William Sigsworth**, A.M.Inst.Pet., on being among the first recipients of the award of the George Medal for conspicuous gallantry.

INSTITUTE SCHOLARSHIP.

The Council has awarded an Institute Scholarship of £40 per annum tenable at the Imperial College of Science and Technology to Mr. **Geoffrey Jardine Clarke**, Stud.Inst.Pet. (Royal School of Mines), for the year 1940–41.

PAPERS FOR THE *JOURNAL*.

Owing to the war, work on a number of papers which were under preparation for the *Journal* or meetings of the Institute has had to be suspended. The Honorary Editor and the Publication Committee will therefore welcome the submission by members of papers suitable for publication in the *Journal*. The condition on which papers are printed is that they shall be approved by a referee appointed by the Publication Committee and subsequently by the Censorship Bureau of the Ministry of Information.

ARTHUR W. EASTLAKE,
Honorary Secretary.

INSTITUTE MEETINGS.

It has not been possible to arrange the customary programme of meetings of the Institute in London for the first part of the 1940–41 Session. Informal meetings or luncheon meetings may be held, and separate notification of these will be sent to all members residing in the London and Home Counties area as and when they are arranged.

If circumstances permit, preprints of papers to be published in the *Journal* will be circulated for written discussion.

BRANCH NOTES.

Meeting of U.S.A. Members.

Members of the Institute in U.S.A. are holding their Annual Dinner and Meeting at the Stevens Hotel, Chicago, on Wednesday, 13th November. Mr. T. A. Boyd, Director of Research, General Motors Corporation, will be the speaker.

STANLOW BRANCH.

The following programme of meetings of the Stanlow Branch has been provisionally arranged for the first part of the 1940–41 Session :

Wednesday, 23rd October — **Friction and Lubrication.** by J. E. Southcombe.
Wednesday, 13th November — Subject to be arranged.
Wednesday, 11th December — **Automatic Instruments in Petroleum Refining,** by D. J. Pull.
January 1941 — Address by the President, Prof. A. W. Nash.

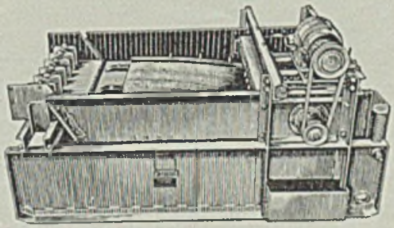
Informal meetings, at which short lectures, followed by discussion, will be the rule, will be arranged as opportunity offers.

Enquiries regarding the activities of the Stanlow Branch should be addressed to the Hon. Secretary : Mr. J. C. WOOD-MALLOCK, Lobitos Oilfields Refinery, Ellesmere Port, Cheshire.

BIRMINGHAM STUDENTS' SECTION.

The Annual General Meeting of the Birmingham Students' Section was held at the University on 9th October. The following were elected officers and committee of the Section for the year 1940–41 :

Chairman : V. D. DAFT.
Vice-Chairman : H. A. CHEETHAM.
Hon. Secretary and Treasurer : Dr. A. H. NISSAN.
Committee : I. A. WILLMOTT, G. BUXTON, J. R. OVER.



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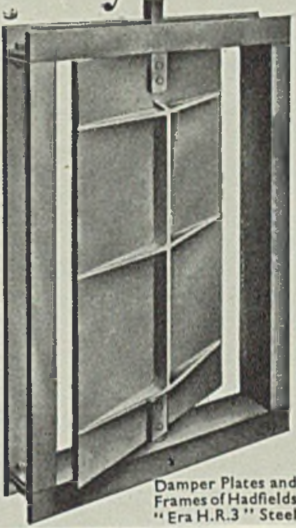

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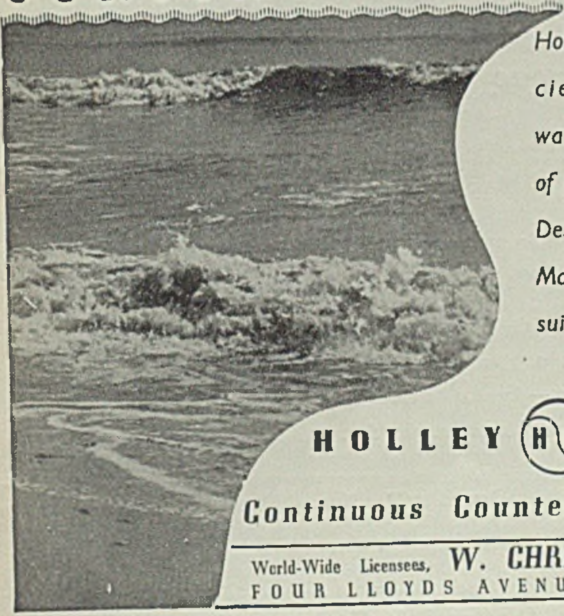

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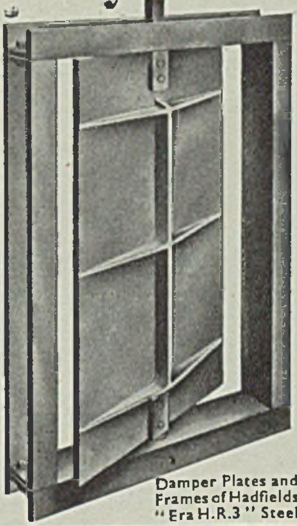

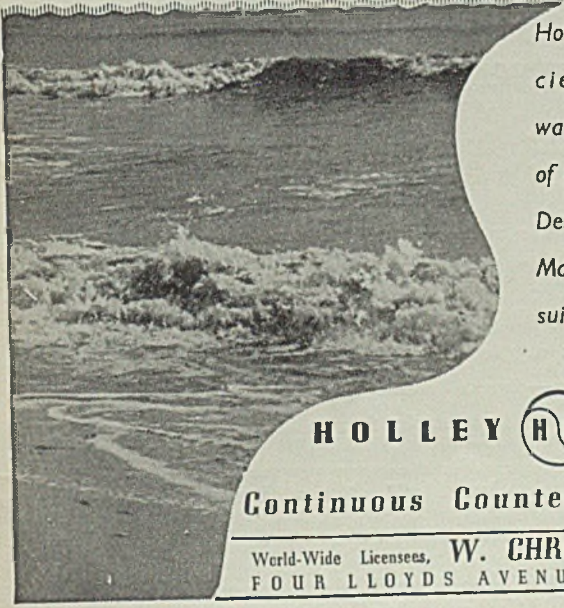

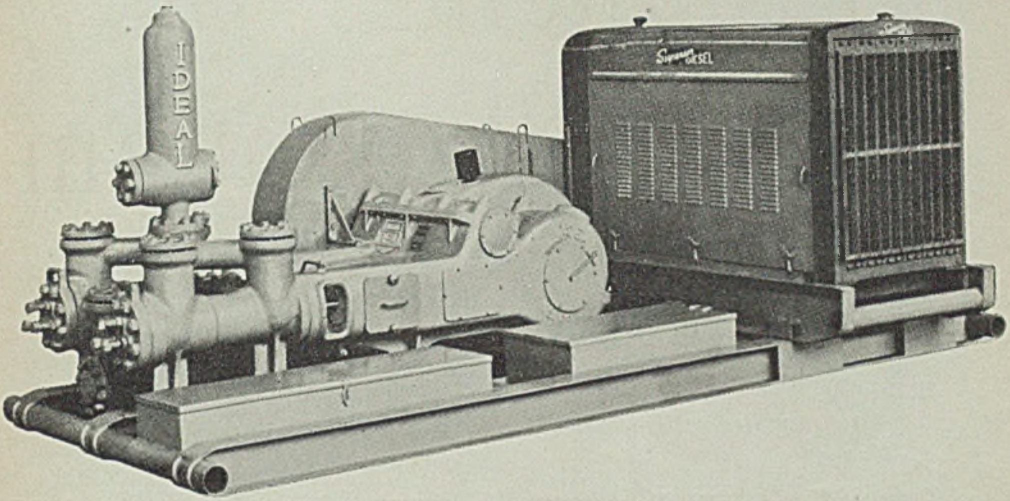
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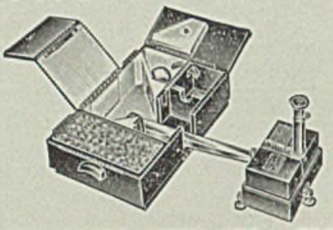

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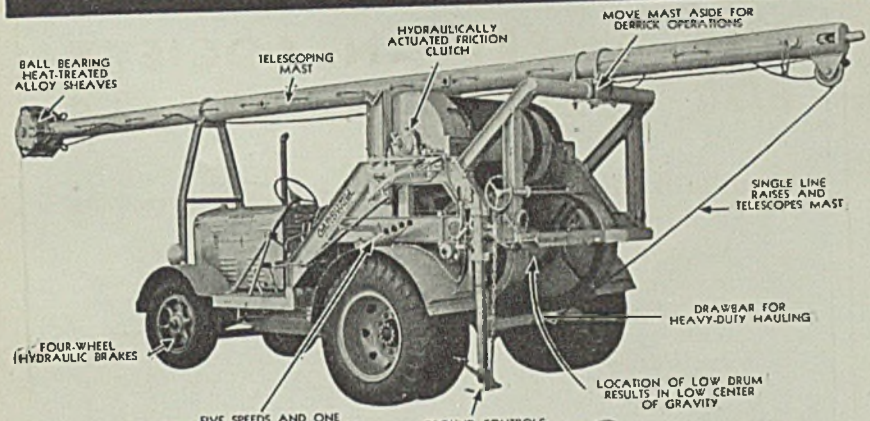

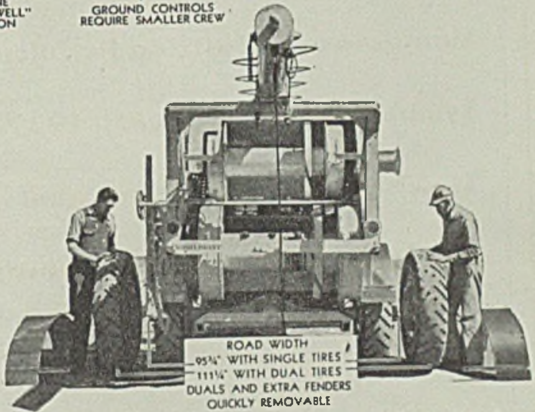

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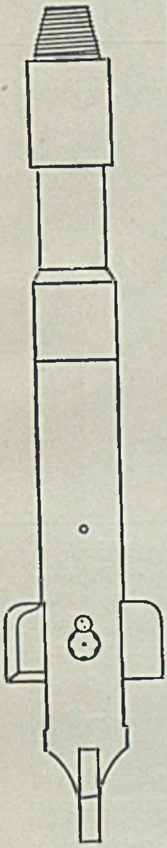

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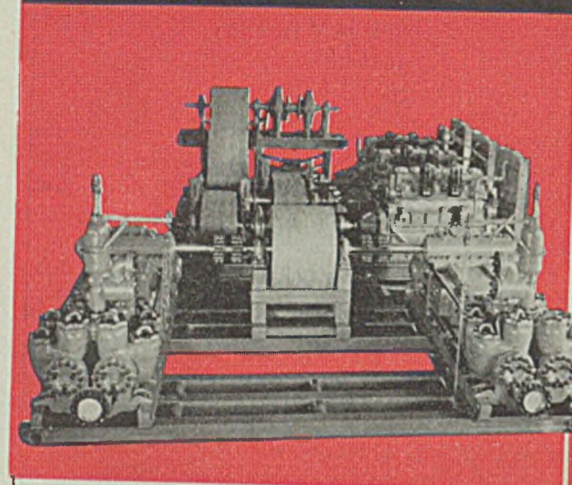

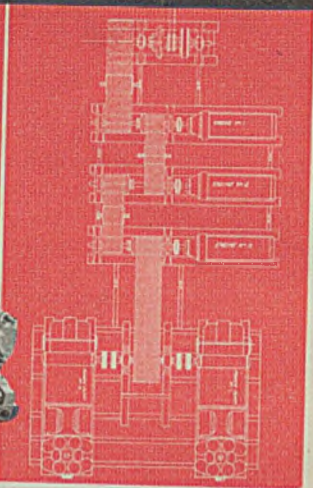

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