

SOME APPLICATIONS OF ASPHALTIC BITUMEN IN INDUSTRY.*

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SIR LEONARD WOOLLEY, in the course of his excavations at Ur of the Chaldees, succeeded in unearthing a remarkably interesting mass of asphaltic bitumen which probably dates back to the year 3200 B.C. Its size and shape suggested that it had been at some time contained in a basket, and we may reasonably conclude that it represents the surplus remaining after a workman had finished some waterproofing or cementing operation.

It is known that naturally occurring deposits of asphaltic bitumen were available, and that this material was extensively used in early Biblical times for a variety of purposes. This basketful of asphaltic bitumen from Ur may therefore be regarded as illustrating one of the very early industrial applications of asphaltic bitumen.

Since those very early times the characteristic properties of asphaltic bitumen have created a demand which could only be met by distilling suitable crudes on a large scale, and in the development of this process of distillation the petroleum industry has displayed such commendable enterprise that the modern distilling plant bears very little resemblance to the conventional still of the early text-books. The modern still used for the manufacture of asphaltic bitumen is, in fact, an instance of the application of exact science to industry, the engineer and physicist having joined hands to produce a highly efficient plant designed, constructed, and controlled strictly along scientific lines.

Asphaltic bitumen is now manufactured in plants operating under vacuum, suitable crudes or topped crudes being distilled to yield products graded according to their penetrations. A typical plant includes a pipe-heater, an evaporator and a fractionating column with the usual barometric condenser and the necessary heat-exchangers. In the vacuum plant the pressure at the top of the fractionating column may be of the order of 30 mm. of mercury, whilst the temperature of a particular topped crude passing to the evaporator was about 350° C., as compared with 400° C. in the case of an atmospheric plant for the production of the same grade (45 penetration). This vacuum process shows a decreased fuel and steam consumption, and yields improved products. The manufacture is controlled by the regular determination of the penetration of the product, and hourly penetration determinations have to be made. For this purpose the standard penetration test is rather lengthy, and a "rapid" modification has been devised. In this modification the apparatus consists of four small containers mounted on a base plate. Each container has the following dimensions :

Height	25 mm.
Internal diameter	12.5 mm.

The four cups are filled in the usual way, and cooled in air for 5 minutes and in water at 25° C. for 10 minutes. The contents of one cup are then

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penetrated. From the penetration figure obtained a time factor is chosen from the following table:

Initial Penetration.	Time Factor.
Nil- 60	5 seconds
61- 90	6 "
91-110	7 "
111-190	8 "
191-215	9 "

The contents of the remaining three cups are then penetrated for this time, and the average penetration is reported.

Although the straight grades find many applications in industry, there are other industries which call for grades which are less susceptible to temperature changes and which retain some measure of pliability at relatively low temperatures. Such grades are produced by "blowing" a soft grade manufactured in the ordinary way. The precise nature of the chemical changes which take place during this "blowing" action has not been established, but it is agreed that the chemical action consists essentially of the removal of hydrogen by the oxygen. This removal of hydrogen probably leads to the formation of more complex molecules, and the properties of the bitumen undergo a fundamental change. The "blown" products show higher melting points for given penetrations or, in other words, they have higher penetration indices. Briefly, "blown" grades are less susceptible to temperature changes, and they are more pliable than the straight grades. It should perhaps be emphasized here that such "blown" grades do not contain any appreciable amount of combined oxygen.

The chemical action resulting from the "blowing" of the bitumen is exothermic, and temperature control is a matter of great importance. The effective use of the air is also a very important consideration in connection with the process. Both of these considerations have been satisfactorily dealt with in the continuous "blowing" processes now in operation. One type of continuous "blowing" plant consists essentially of a vertical vessel with multiple air-jets passing through the base. The level of the bitumen is maintained constant, and the air supply so regulated that maximum use of the available oxygen is attained. With this type of plant, temperature control is greatly facilitated and uniform products are obtained.

The following table gives the properties of "blown" grades of bitumen on the market:

Melting point (R. & B.), ° C.	130-140	110-120	80-90	80-90	70-80
Ductility at 25° C.	1 cm.	2 cm.	3 cm.	3 cm.	4 cm.
Penetration at 25° C.	7-12	10-20	35-45	20-30	25-35

If the straight grades are added to these, a very wide range of closely controlled products is available, and it is not surprising that in one way or another they have found applications in a multitude of industries. It is, of course, only possible to select three or four typical applications for discussion in this paper, but it is hoped that other papers will be forthcoming dealing with specialized applications in greater detail.

PIPE LINING AND COATING.

Manufacturers of steel pipes soon appreciated the suitability of asphaltic bitumen for the protection of both the insides and the outsides of pipes.

Relatively thick coatings are normally used, and it is obvious that such coatings must neither flow when the pipes are exposed to the sun during storage, nor crack at low temperatures in transport or when being laid.

The following special tests have been devised to ensure that the suggested compositions meet these requirements. These tests are known as the "flow test" and the "shatter test."

In the "flow test" a coat of the composition, 5 mm. in thickness, is applied to a flat steel plate. White marks are then painted across the width of the coating. The assembly is supported at an angle of 45° in an incubator maintained at the test temperature. The average displacement in millimetres of the marks after 20 hours is taken as a measure of the flow.

The "shatter test" consists in determining which of a series of steel balls is just capable of causing a 5-mm.-thick coating of the composition to crack when the ball is dropped from a height of 2 metres on to a standard steel plate to the under-side of which the coating under test is adhering. Standard ball-bearings of various sizes are used, and the weight of the ball just capable of cracking the test-piece is reported as the result of the test.

Compositions complying with the following general requirements have given satisfactory results when used as inside coatings to be applied in a molten condition by the rapid rotation of the pipe :

Melting point (R. & B.), ° C.	100-130
Penetration at 25° C.	25- 10
Flow in a 5-mm. layer at 70° C. at 45° inclination after 20 hours	Less than 1 mm.
Shatter test at 15° C.	358 grms.
" " " 0° C.	45 "
Maximum filler content	30% wt.

"Blown" bitumens are used in the preparation of such compounds. The application of thick coatings naturally reduces the effective internal diameter of the pipe, and it may appear that there must be a decrease in the carrying capacity of the coated pipe. Such is not the case, however, and it has been established that the loss of head for the same length of a bitumen-lined pipe is 20 per cent. less than in the case of a similar pipe uncoated. It has been established in practice that for diameters in excess of 20 cm., pipes with a bitumen lining 0.5 cm. in thickness will always have a greater carrying capacity than the same pipe uncoated.

These observations apply to new pipes, and the advantage in favour of the bitumen-lined pipe will obviously be much greater in the case of old pipes, where corrosive products greatly increase the friction and reduce the carrying capacity of uncoated pipes.

Much work has been carried out in connection with the production of satisfactory outside coatings, and highly successful results have been obtained.

In the first place, two special tests for the examination of outside coatings merit attention. It is, of course, essential that a satisfactory outside coating should be non-porous and free from pin-holes. In the case of thin coatings, the condition in this respect can easily be tested by the potassium-

iodide test. In this test the positive pole of a $4\frac{1}{2}$ -volt pocket battery is connected to the metal of the test-pipe. The negative pole is connected to a lead plate, which is pressed against a piece of muslin soaked in potassium-iodide starch solution which lies on the surface of the coated pipe. The current is allowed to pass for one minute, and the lead plate then removed. The existence and positions of any pin-holes are established by the presence of dark-blue spots on the cloth. Thicker coatings can be tested along somewhat similar lines using a high-tension alternating current of low amperage, porous spots being indicated by the occurrence of sparking.

For complete impermeability it is necessary to use coatings of at least 0.5 mm. thickness, and the use of "blown" bitumens is desirable. An apparatus designed for measuring the thickness of the coating consists of a stout, insulated needle mounted at the extremity of a micrometer screw, which in turn is mounted on a stand which can be firmly strapped to the pipe under examination. This needle is connected to one contact point of an indicator lamp. The stand is also fitted with a set screw which is connected to the other contact of the lamp. Contact with the actual metal of the pipe is first established by means of this set screw. To measure the thickness of the coating, it is then only necessary to bring the needle into contact with the bituminous surface, read the micrometer, and then screw down the needle through the coating until it touches the metal, when the micrometer is again read, the difference between these two readings giving the thickness of the coating. The contact between the needle and the pipe is accurately established, since the needle completes the circuit and the signal lamp is lighted.

It has been found necessary to incorporate micro-asbestos in such compositions and a suitable composition consists of 70 per cent. of "blown" bitumen and 30 per cent. micro-asbestos. The "blown" bitumen must be so chosen that the composition does not flow more than 1.5 mm. at 75° C. or 40 mm. at 100° C. when tested as described above. In applying these coatings, it is usual to paint the pipe with red lead and apply a priming coat of a solution of a "blown" bitumen similar to that used in the composition itself. Such pipe enamels may be further protected and reinforced by being wrapped with coir fabric, a material which is extraordinarily resistant to the various disintegrating influences.

JOINT-FILLING COMPOUNDS.

Bituminous joint-filling compounds are used in considerable quantities for a variety of purposes, but a feature common to all applications is that the filling compound must prevent water penetrating into the joints without the separate parts of the structure being rigidly jointed. Asphaltic bitumen meets these requirements very satisfactorily, since it resists even aggressive waters, whilst, on account of its plastic nature, it allows a certain amount of movement of the parts without damage to the joints.

For some purposes it is desirable to add filler, and the following figures show the influence of the nature of the filler upon the stability of the mixture:

Mixture of : 55% asphaltic bitumen (50-60 penetration), 45% filler.	Melting point (R. & B.), ° C.	Height of fall of 50- gm. weight at 0° C. to cause cracking, cm.
Quartz	55.5	160
Limestone	55.5	170
Powdered shale	57.0	200
Micro-asbestos	61.0	700

The increase in shock resistance conferred by a fibrous filler is quite striking.

As might be expected, "blown" bitumens also show good shock resistance, as shown by the following results :

	Melting point (R. & B.), ° C.	Penetration at 25° C.	Shatter test at 0° C. Height of fall 2 metres.
Normal asphaltic bitumen .	57.0	45	36 grms.
"Blown" asphaltic bitumen	85.5	40	357 "
"Blown" asphaltic bitumen	93.0	36	761 "

In certain cases it is necessary that jointing compositions should be able to accommodate movements of concrete masses due to subsidence or changes in temperature. These conditions call for a measure of ductility and satisfactory adhesion. These features may be studied by means of the special apparatus shown in the slide.

A joint 15 mm. wide between two concrete blocks is filled with the composition under test. The concrete blocks are cast with grooves, by means of which they can be fixed in the clamping device of the apparatus. One half of this clamping device is fixed, whilst the other is free to slide along a guide-rail. This free half of the clamp is attached to the free end of a bronze rod. This rod is surrounded by an electrically heated oil-bath. In carrying out a test, the oil-bath is heated at a controlled rate, and the expansion of the rod is transmitted to the free half of the clamp. In this way the blocks are caused to approach each other, and the jointing composition is slowly compressed and partly expressed from the joint. On cooling the oil-bath, the reverse action takes place, and the jointing composition is stretched. The apparatus works smoothly and slowly, reproducing practical conditions.

In an actual test the joint is narrowed by 3 mm. during about 3 hours, and then stretched through 3 mm. in about 10 hours, the whole process being repeated at least six times.

The following mixtures gave satisfactory results when tested in this way :

- A. 50 parts fluxed "blown" asphaltic bitumen
(Melting point (R. & B.) 51° C.)
(Penetration at 25° C., about 140);
50 parts powdered asbestos (Asbestine).
- B. 42 parts by wt. asphaltic bitumen, 300 penetration;
58 " " " fine sand;
11½ " " " asbestos fibres (2-4 mm.).

The construction of satisfactory joints is of special importance in connection with hydraulic engineering, and protective revetments of stone or concrete on coasts, rivers, canals and reservoirs offer great scope for use of bituminous joint-filling compounds.

The purpose of the joint is primarily to prevent the attack of the water on the underlying soil, but in the case of stone revetments, the mechanical resistance of the revetment is also greatly increased by the cementation of the individual stones. It is also of great importance that such stone revetments should be able to follow slow settlements of the soil without fracture. Bituminous jointings meet this particular requirement, and, in addition, they offer satisfactory resistance to corrosive waters.

In hydraulic work the surfaces to be protected are usually sloping, and the resistance to flow shown by the jointing materials becomes of outstanding importance. On the other hand, high ductility and shock resistance are not essential.

A very simple test has been worked out for the determination of the flow resistance of such jointing compounds.

In this test emphasis is laid on the imitation of practical conditions, and suitably shaped slabs of concrete are separated by distance pieces and bolted together to form imitation joints. These are 5 cm. deep and 1 cm., 3 cm., or 5 cm. wide. The slope is determined by the practical conditions to be met, but is usually 1:1. The apparatus shown in the slide is arranged for the simultaneous examination of three compounds. When the assembly has been completed, the original positions of the compounds are marked by rows of white dots, and the whole construction is maintained at the maximum temperature expected in service. The amount of flow taking place is indicated by the displacement of the white dots, and measurements are made at regular intervals.

It has been found that it is essential to incorporate fibrous fillers in bituminous jointing compounds to be used on slopes even in temperate climates, and short-fibred asbestos has given most satisfactory results.

Slag wool and cotton have also been used with some success, but special care is necessary in handling and applying these materials.

Mixtures of the following two types have given satisfactory results in practice:

(1) Mixtures containing a relatively high percentage of bitumen of medium hardness, sand, and a small percentage of short-fibred asbestos.

These mixtures can be poured at 180–200° C.

(2) Mixtures containing a relatively low percentage of soft bitumen, sand, filler, and a small percentage of asbestos.

These mixtures have to be trowelled.

Both types adhere well to stone, but type (2) mixtures are rather more ductile at low temperatures.

In general it is permissible in hydraulic work to use a harder bitumen or a higher percentage of mineral filler with a soft asphaltic bitumen than would be necessary in the case of road-work, which naturally calls for greater shock resistance and higher ductility.

HYDRAULIC WORKS.

One of the very oldest applications of asphaltic bitumen seems to have taken a fresh lease of life during the last few years. I refer to the use of asphaltic bitumen in connection with hydraulic works. The protection and waterproofing of river- and canal-banks, dams, reservoirs, swimming-pools, coast works, etc., give rise to a number of interesting problems, and have inspired many investigations.

This is a very large subject, but it will suffice in this paper to consider one or two typical works in some detail.

The waterproofing of the Ghrib Dam, Algeria, provides an interesting and instructive example of bituminous hydraulic work. This work entailed the waterproofing of the upstream face of a rock-filled dam 65 metres in height. The slope in this case steepened from 1 : 1 at the foot to 1 : 0.67 at the summit.

The waterproof mask was built up of the following courses :

- (1) A drainage layer (8 cm.) of lean cement concrete.
- (2) Cutback tack coat.
- (3) 6 cm. asphaltic concrete.
- (4) Tack coat 200 penetration.
- (5) 6 cm. asphaltic concrete.
- (6) Cement mortar tack coat.
- (7) A protective and heat-insulating layer (10 cm.) of concrete slabs suspended from the crest of the dam by a continuous reinforcement.

The asphaltic concrete aggregate used was composed of :

30 per cent.	weight	12/25 mm.	limestone	chippings.
15	„ „	„	5/12 mm.	„ „
25	„ „	„	0/5 mm.	„ „
20	„ „	„	fine sand.	
10	„ „	„	filler.	

Composition of Mix.

92 per cent. aggregate.

8 „ „ asphaltic bitumen, 20/30 penetration.

A striking feature of this work was the ingeniously designed steel bridge which carried an electrically driven 5-ton roller, and also the working platform from which the mix was spread.

The success of the work may be judged from the fact that percolation through the revetment under a head of 32 metres of water over an area of 8000 sq. metres amounted to only 1.3 litres per minute.

Experience has shown that in the construction of bituminous revetments on steep slopes it is essential to secure a good bond between the bituminous layer and the underlying surface. This consideration is at least as important as the composition of the bituminous layer, if flowing is to be prevented, and the application of the tack coat is perhaps the most delicate operation involved. Either an excess or a deficiency of tack coat will result in slipping of the bituminous layer. In view of this experience, it is recommended that a reinforcement should be introduced between the two

layers of asphaltic concrete. This reinforcement could be anchored to the crest of the dam.

A very interesting experimental "under-water" application of mastic was recently carried out at Scheveningen (Holland). The work was carried out on one of the groynes on the beach. These groynes, which are extensively used in Holland for coast protection, extend for a distance of about 200 metres from the shore into the sea.

In building such a groyne, a core of fascines is first constructed. This, in turn, is covered with straw, on which is laid a bed of gravel. This serves as a foundation for a crown course of basalt blocks, which are enclosed by stakes on either side. Beyond these stakes there is a pitching of basalt blocks at least 1 metre in depth on each side of the groyne. These pitchings serve the purpose of loading and protecting the fascines. Storm damage is considerable, and consists mainly in the removal of the pitching. It was thought that this damage could be prevented if the voids of the pitching were filled with mastic, and the present indications from the trial are very promising.

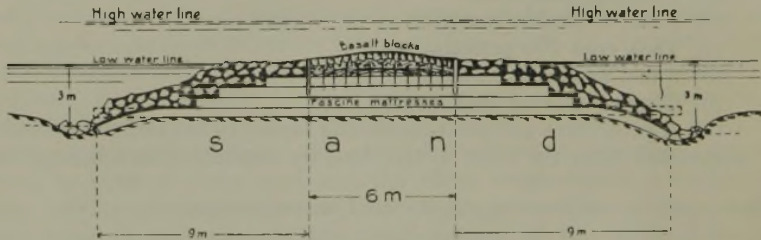


FIG. 8.

CROSS SECTION OF BREAKWATER, SCHEVENINGEN.

The length of groyne for the trial was so chosen that the pitching was almost covered at low tide, with the high-tide level at least $1\frac{1}{2}$ metres above, so that the crown of the groyne is submerged twice daily under at least 1 metre of water. The mastic used had the following composition :

- 70 per cent. fine sand.
- 10 " " filler.
- 20 " " asphaltic bitumen, 60/70 penetration.

This mastic at 180° C. was conveyed in hand-barrows and tipped directly into the voids, until it was judged that they had been completely filled. The consumption of mastic amounted to about 600 kg. per square metre. The maximum depth of water through which the mastic was poured was 3 metres at the toe of the pitching, about 12 metres from the centre-line of the groyne.

It is obvious that no real adhesion can be expected under such conditions, but in this type of work it is considered that adhesion is not necessary, and it is hoped that so long as the voids are filled by a permanently plastic yet stable mass, the structure will be able to resist the attack of the waves.

In spite of the high consumption, the process will, if reasonably successful, show a marked economy over present maintenance costs.

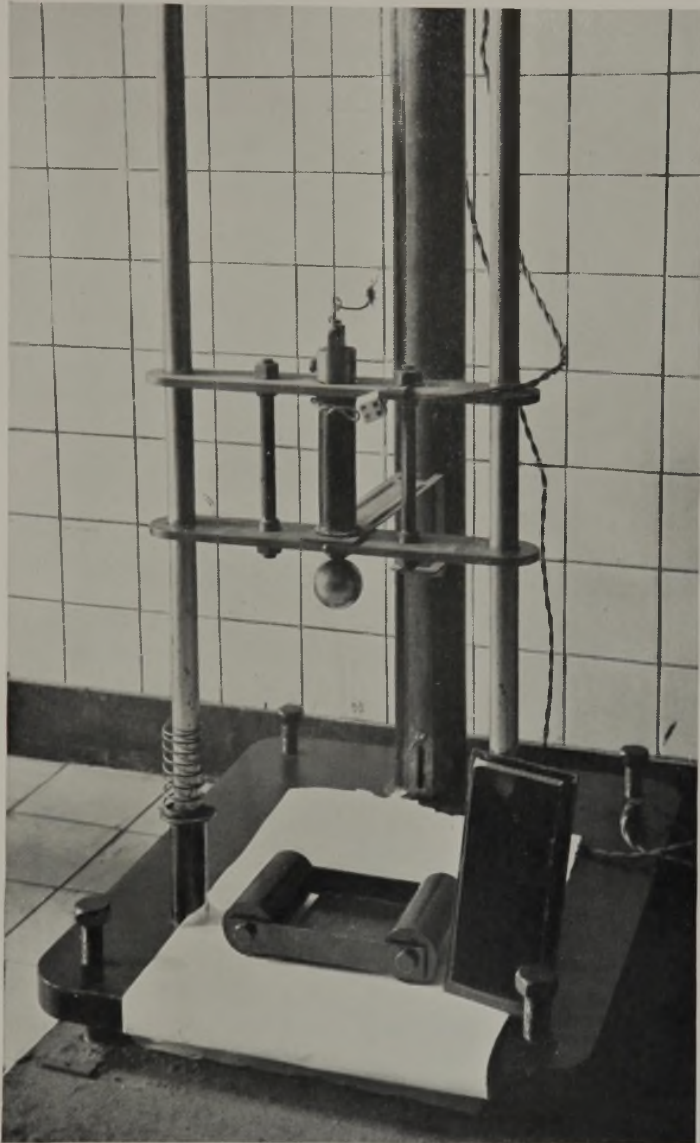


FIG. 1.
DETAIL OF THE SHATTER TESTER, CLEARLY SHOWING THE WAY IN WHICH THE
PLAQUE IS SUPPORTED

[To face p. 58.]

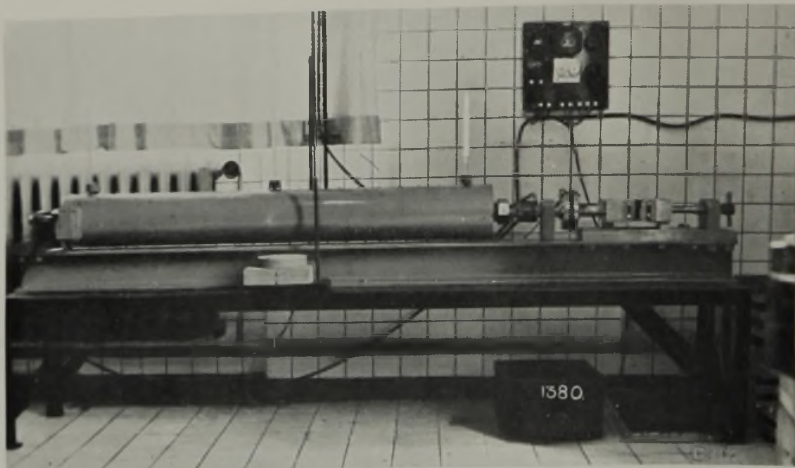


FIG. 2.
APPARATUS FOR COMPRESSION AND EXPANSION TESTS.

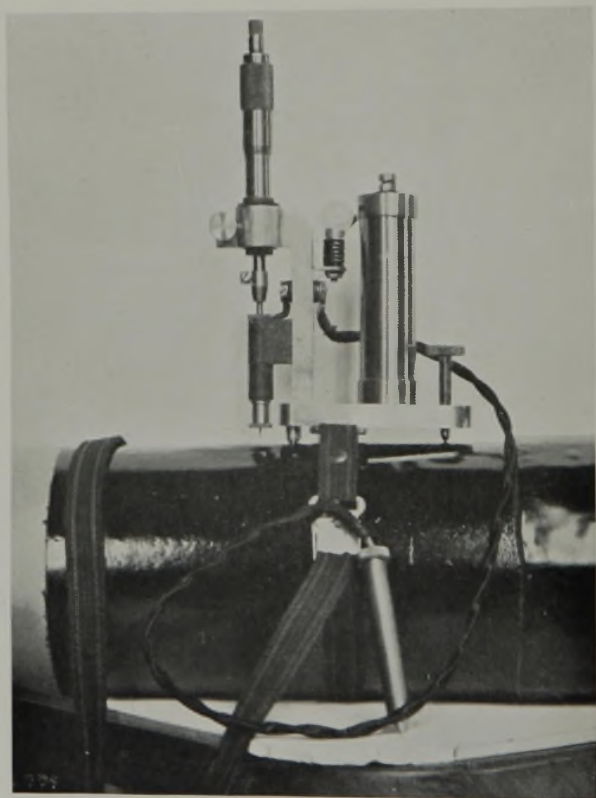


FIG. 3.
APPARATUS FOR DETERMINING THE THICKNESS OF PIPE COATINGS.



FIG. 4.
CONTINUOUS BLOWING PLANT.



FIG. 5.
ASSIUT DAM, SHOWING EMULSION BEING INJECTED.



FIG. 6.
SCHEVENINGEN BREAKWATER.

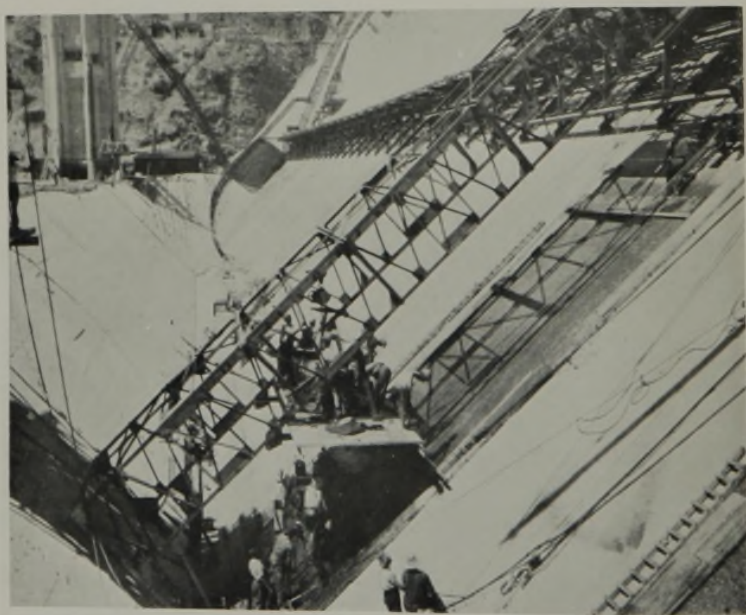


FIG. 7.
GHRIB DAM. STEEL WORKING BRIDGE.

[To face p. 59.]

CLAY EMULSIONS.

Dispersions of bitumen in clay suspensions find many applications in industry. Such dispersions are produced by mechanical means, using such materials as bentonite as the emulsifying and stabilizing agents. The resulting emulsions are thixotropic pastes containing 50–60 per cent. of bitumen. They differ from the soap type of emulsions in many important respects, one interesting feature being that the bitumen particles are nearly all cigar-shaped, and not spherical. The emulsions are extremely stable to both mechanical and chemical action, and they do not coagulate to films of bitumen until the water in the emulsions has been completely evaporated. The residual films are supported by the clay skeleton, and are surprisingly resistant to flow under heat. Such emulsions are widely used in the manufacture of bituminous paper and boards, and in various forms of protective films. The manufacture of clay emulsions presents many problems and difficulties, and has not yet been reduced to a systematic process. While various clays may be used, the bentonites are very efficient emulsifiers, producing satisfactory emulsions with 3–4 per cent. of clay (calculated on the bitumen).

The first step in the manufacture is to prepare a suspension of the required amount of clay in water; this is known as the clay slip. This is usually done by first preparing a concentrated suspension containing perhaps 15 per cent. of bentonite in order to increase the effect of agitation. This concentrated suspension is then diluted with further water to the required concentration. Before the clay slip can be used as an emulsifier, it is usually found necessary to add an electrolyte, which has the effect of flocculating the clay to some extent.

The principle involved in the large-scale preparation of clay emulsions is very simple, but in practice the process is very uncertain, and it is quite impossible to describe any precise procedure. In general terms the molten bitumen is poured continuously and simultaneously with the clay slip into a suitable form of mixer fitted with an efficient stirring device. The bitumen is pulled out into thin threads, which ultimately break into the characteristic cigar-shaped particles. The emulsion during mixing is maintained at a temperature approximating to the melting point of the bitumen. With bitumen at 120° C. the clay slip may be maintained at about 45° C.

The manufacture of paper consists essentially in the removal of water from a uniform layer of fibre deposited from a very dilute suspension of fibre in water. The preparation of the suspension involves the disintegration in a beater of the particular fibrous material used.

In view of the stability of these clay emulsions to mechanical and chemical action, it has been found possible to add them direct to the pulp in the beater and proceed to manufacture bituminous paper by the ordinary paper-manufacturing process, thus eliminating the more usual second impregnating process. The emulsified bitumen passes through the whole process with the fibre, and is finally remelted to form a waterproof coating on the fibres in the calendering process, which is primarily designed to give the paper the necessary glazed finish.

Stable clay emulsions are easily handled, and it is not surprising that they

are used for a great variety of purposes. Perhaps the latest application is in connection with the protection of wooden structures against incendiary bombs.

Wooden floors can be protected by a layer of sand, but this is not very satisfactory, as it is easily displaced. A more satisfactory and permanent method is to coat the floor with a bituminous mastic. This, however, only deals with part of the problem, as the shower of sparks from the bomb may ignite the walls of the building, which cannot very well be covered with mastic. It has been found that wooden walls can be effectively protected by applying a 1-cm. layer of a mixture of 1 part clay emulsion and 4 parts sand. This mixture can be easily applied and worked into cracks and corners. Such a coating gives very effective protection, but there is a risk that prolonged exposure to flames would destroy the adhesion of the coating to the wall, and for complete protection it is advisable to reinforce the coating by means of wire netting, which can be nailed to the wall.

This mixture of emulsion and sand can equally well be used for the protection of wooden floors, and the mixture obviously provides an effective and simple means of protection for attics and lofts.

The Shellperm Process constitutes another very interesting specialized application of a bitumen emulsion.

In this process a very fine emulsion is pumped under moderate pressure into suitable porous soils, and caused to break when it has reached the desired location. The emulsion is used in a diluted form containing about 30 per cent. of asphaltic bitumen, and, as used, it is practically as thin as water. As the specific gravity of the asphaltic bitumen differs little from that of water, the emulsion can be made to flow, without sedimentation, through permeable soil containing fine pores in the same way as water percolates. Under the same conditions the emulsion moves only a little more slowly than water through the soil. The emulsion can therefore be pumped through injection tubes into the soil under treatment at comparatively low pressure. Under these conditions the ground-water in the pores of the soil is replaced by the emulsion without any appreciable amount of mixing taking place. As injected, the emulsion has no particular effect upon the soil, but by the addition of suitable "coagulants" to the emulsion it can be made to break at the expiration of any predetermined period, which may vary from a few minutes to several days. The coagulated particles of bitumen then clog the pores of the soil, rendering it impermeable.

In this process the concentration of the emulsion injected, and thus the amount of bitumen put into the soil, and also the time of flocculation, are under control.

The size of the pores in the soil to be treated is naturally an important consideration. In a coarse sandy soil the dispersion will naturally penetrate more easily than in a fine-grained soil. Care must therefore be taken that the particle size of the disperse phase is adjusted to the soil porosity in accordance with the required depth of penetration. The particle size can be controlled during the manufacture of the dispersion, and any coarse particles removed by careful screening or filtering.

In applying this process, injection pipes are driven into the soil at suitable distances from each other, and to such a depth that their open or perforated ends reach the porous layer in which an impervious screen is to be formed. The required quantity of the dispersion to which a suitable coagulant has

been added is then poured or pumped into each of these pipes, and allowed to flow into the surrounding porous soil, filling the voids of the soil, until dispersion issuing from the various pipes joins up forming a continuous impregnated layer. The essential feature of the process is that the dispersion can then be caused to coagulate in accordance with the known conditions and the particular coagulant used.

In addition to rendering the soil impermeable, the deposited bitumen has a marked stabilizing effect.

The following two examples will serve to illustrate the application of this process :

(1) *Sealing the Bottom of a Building Pit.*

A building pit approximately 70 ft. long \times 22 ft. wide \times 14 ft. deep was suddenly filled by ground-water, which welled up as the result of a thin clay layer being disturbed by the driving of piles. The stratum of sand at the level of the foot of the sheet-piling was made satisfactorily watertight by sixty injections of emulsion.

(2) *Assiut Barrage on the Nile.*

With a view to strengthening the barrage, the foundations had to be extended. For this purpose a coffer-dam made by driving sheet-piling had to be constructed on each side of the barrage. In the river this piling reached 4 metres below the concrete foundation slab, but where the piling converged upon the dam it was naturally not possible to drive piles through the foundation, and the piling was therefore continued over the top of it. Owing to the permeable nature of the subsoil, water would have seeped into the coffer-dam from under the foundations. It was therefore necessary to render the subsoil impermeable at this point to the normal depth of the sheet-piling. Holes were therefore drilled through the concrete foundation, and bitumen emulsion was injected at low pressure to form an impermeable screen, 30 metres long, 5 metres wide and 4 metres deep, under the foundation. Practically complete impermeability was achieved.

The process can be operated only in rather porous soils containing few grains finer than $1/30$ of an inch.

As the coagulated bitumen is plastic, this process offers certain advantages over the use of cement slurries.

Large quantities of asphaltic bitumen are used in the production of roofing-felts and floor-cloths. It also finds many specialized applications in the electrical industries. These applications all present technical problems of great interest. If to these problems we add the many others connected with the manufacture and application of emulsions and with the chemistry and physics of asphaltic bitumen itself, we can surely anticipate a long list of valuable papers which we hope will be presented before a Bituminous Materials Group of this Institute.

ACKNOWLEDGMENT.

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THE INSTITUTE OF PETROLEUM.

INAUGURATION OF ASPHALTIC BITUMEN GROUP,
DECEMBER 13th, 1938.

THE one hundred and eighty-fourth General Meeting of the Institute of Petroleum was held at the Royal Society of Arts, John Street, W.C.2, on Tuesday, December 13th, 1938. LIEUTENANT-COLONEL S. J. M. AULD, O.B.E., M.C., D.Sc., the President of the Institute, occupied the Chair.

THE PRESIDENT said that the present meeting was the inaugural meeting of the Asphaltic Bitumen Group. The Council of the Institute was looking to the Groups, two of which had now been formed, to carry into fulfilment a good many of the hopes that had been founded on the broadened basis of the Institute. He thought the excellent attendance at the present meeting showed that the formation of Groups for the study of individual problems was the right step to have taken. Decentralization of that kind would not have any adverse effect on the General Meetings of the Institute. It would only strengthen the Institute and at the same time provide an opportunity, which had previously been lacking, for members with particular interests in common to meet together and discuss their own problems.

The Council had invited Mr. J. S. Jackson to be the first Chairman of the Asphaltic Bitumen Group, and he would ask Mr. Jackson to explain his ideas with regard to its operations.

MR. J. S. JACKSON, B.Sc., F.I.C., said the first announcement he had to make was that there would be no additional fee charged to members of the Group, but it was proposed to restrict membership of the Group to members of the Institute. The meetings would be held at the Institute's premises, in the Adelphi. Meetings would be held on any technical subject of interest to those who were primarily concerned with asphaltic bitumen, its manufacture, its properties, its applications and so forth. Those meetings might be small intimate meetings or joint meetings with the Roads and Building Section of the Society of Chemical Industry, or meetings with papers presented to the full Institute. He should perhaps emphasize that it was not proposed to deal with subjects which would be more suitably dealt with by the Roads and Building Section of the Society of Chemical Industry. It was hoped to organize visits to places of interest to those engaged in the manufacture or applications of asphaltic bitumen, and such visits might be combined with short papers. The response had already been very encouraging, about fifty members of the Institute having expressed a desire to become members of the Group. Many of those who had expressed their desire to join the Group had forwarded suggestions as to subjects for discussion, and he wished to thank them for the interest they had shown, to invite further suggestions and to express the hope that volunteers would come forward to present papers. Such papers need not necessarily deal with the results of researches; a very interesting evening could be spent, he thought, in the discussion of an interesting observation

made or a difficulty encountered in some particular industry. It was hoped that members of various industries would present papers of either general or particular interest. If the present meeting was any indication of the future of the Group, he was sure the Group would be a very active and successful one.

If anyone present had any suggestions to make or any questions to ask at the moment, he would be glad to hear them.

As no questions were raised, MR. J. S. JACKSON then presented the following paper: "Some Applications of Asphaltic Bitumen in Industry." (See pp. 51-61.)

The paper was illustrated with lantern slides and a cinematograph film.

DISCUSSION ON "SOME APPLICATIONS OF ASPHALTIC BITUMEN IN INDUSTRY."

THE PRESIDENT said he was sure everyone present would agree that Mr. Jackson's splendid paper had given the Asphaltic Bitumen Group a very good start. He had listened to the paper with the greatest interest, and he hoped that he would have many opportunities of attending meetings of the Group and learning more about the asphaltic bitumen side of the subject of petroleum.

DR. P. E. SPIELMANN congratulated the inaugural meeting of the Asphaltic Bitumen Group. As there seemed to be about two-thirds of the audience usually attending an ordinary meeting of the Institute, the size of the meeting was a very good augury for the success of the Group. The paper had set a standard which the Group would do well to attain in the future. The well-selected miscellaneous matters which the author had put before the meeting had resulted in a fascinating evening and one which they would remember. It was especially good for anyone like himself, whose mind had been directed rather to the road applications of bitumen, to have attention directed to the numerous other uses to which bitumen was being put.

With regard to the blown bitumen, which was looked upon favourably in Holland but with suspicion in this country from the road point of view, he would ask the author how successful the concrete jointing to which he had referred really was, because blown bitumen was considered to be easily oxidized and therefore to be an undesirable substance to use on roads.

He was interested to learn that micro-asbestos has come into its own; he believed it was too expensive to use in roads, but obviously it was a material which was especially suited for asphalt mixtures which were subject to particular stresses.

He congratulated the meeting and the Author on one another.

MR. D. C. BROOME said that there was a vast number of applications of bitumen and it had been impossible for the author to deal with more than a few of them in his paper, but he thought the examples which the author had chosen were particularly interesting. Personally he was especially interested in the first application mentioned in the paper, namely, the question of pipe linings, and two points had occurred to him in that connection. The author had shown a slide illustrating the effect of different types of filler, and he thought it was of some interest to note that the situation was affected not only by the type of filler but also by the fineness of the filler. He had had a rather interesting case of that a year or two ago, when he had had to deal with the preparation of a large consignment of pipe-lining composition that was to be used for lining steel water mains in Penang. Owing to the peculiar conditions obtaining there, a special specification for the compound had to be worked out in the laboratory, and a compound was eventually arrived at which had a softening point of 170° C. That specification was duly passed over to the factory and the factory manufactured a large batch of the material. Samples were taken for checking purposes and it was found that they agreed exactly with the specification as far as composition was concerned, but the softening point, instead of being 170° C., was 130° C. Further investigation showed the trouble to be due to the fact that, in order to assist in getting the high softening point of 170° C., a very fine filler was used, and the plant conditions were such that, instead

of the great fineness of the filler being utilized, partial coagulation or clotting of the filler took place, so that it behaved as a much coarser filler. The whole batch was put back into the plant, and, by still further and more careful agitation, it was found possible to break down the filler without any question of overheating or anything of that kind. Samples taken at intervals gave gradually increasing softening points, until finally the figure of 170° C. was obtained.

His mind also turned to those conditions where the filled bitumen compounds, in the ordinary sense of the word, were not quite sufficient. Sometimes a greater thickness of material was required, and for that purpose one generally used the other compounds to which the author had referred, containing a relatively small proportion of bitumen and a relatively large proportion of mineral matter of one sort or another. That came into the picture particularly in the case of the lining of concrete pipes and had some considerable application where special liquors had to be dealt with, such as sewage. It also helped in the question of speed of production of those pipes. With some of the modern methods of producing concrete pipes it was possible to apply asphalt linings to a thickness of as much as $\frac{1}{4}$ in. immediately on to the green concrete, so that one could manufacture a concrete pipe, line it with asphalt and strip the mould, the whole process occupying about three minutes. That was, of course, very useful in the case of high-speed production work.

With regard to Dr. Spielmann's reference to the question of expansion jointing for concrete work, he also had found micro-asbestos very useful in connection with that kind of work. He had had a rather interesting case not long ago in connection with the foundation work of the new University of London building in Bloomsbury, where large masses of concrete were being placed for foundations and it was desired to provide jointing between the sections of that concrete without interfering with the works. Eventually the following method was adopted: as soon as one section of the concrete had been placed and allowed to stand long enough to enable the shuttering to be removed, the end face of that section was covered with a layer of cold mastic asphalt, manufactured with an emulsified bitumen, micro-asbestos and a certain amount of other mineral material. That was trowelled on in the same way as one applied hot mastic, but, it being emulsified and applied cold, perfect adhesion with the green concrete was obtained. As soon as it was in position the next section of concrete was poured straight up against that cold mastic, with perfect success so far as could be seen. That method had been used once or twice on other occasions, but he thought the case to which he had referred was the largest example of its use.

Mr. J. McCONNELL SANDERS said he thought it must have struck many of those present who had heard the author's excellent account of the modern applications of bitumen, that the very ancient peoples must have acquired a considerable amount of practical knowledge in the application of the material to their requirements although obviously they could not have had available anything approaching the modern resources of science and technology. It was astonishing to him—and he thought it must be to anyone who had read Dr. Forbes' book on the applications of bitumen in antiquity—how nearly modern practice approached in its final results the methods utilized by ancient peoples who had a similar object in view.

The use of chopped straw or similar fibrous material to prevent the soft bitumen from slipping on steeply inclined surfaces had its modern counterpart in the use of short-fibred asbestos. The bitumen-caulked cradle of the infant Moses was but a foreshadowing of that more elaborate application wherein bitumen formed an important ingredient of the material used in building a modern dam.

It is curious that in Mexico, which is probably the greatest producer of asphalt in the world to-day, there is little evidence that its ancient inhabitants utilized the deposits of naturally occurring asphaltic bitumen in their arts or industries. It is supposed that bitumen was used by the Aztecs as a form of incense burned to their gods, but it is difficult to understand how a material containing from 5 to 8 per cent. of sulphur could have been pleasing to the nostrils either of the gods or their worshippers!

There was one point which he wished to make with regard to the future work of the Group and that was its relation to those other industries in which bitumen was already, or could be, utilized. The manufacturers of the various forms of commercial bitumen were expending considerable time and energy in making their products suitable

for a great variety of commercial purposes, and in devising new applications for their material, but in many cases they lacked a corresponding amount of knowledge regarding the advances which had been made in the treatment of the materials with which their product would eventually be used.

It seemed to him that it would be extremely useful if the representatives of these other industries, for instance the paper and concrete makers, the manufacturers of paints, engineers and members of the building trades, could attend the meetings of the Group and collaborate with its members in discussing any modifications which could be made, either in the bituminous product or in the material with which it was designed to be used.

MR. E. LAWSON LOMAX said that it might be thought, from what had been said about bitumen that evening, that it was a definite chemical compound, but in fact very little was known about the composition of bitumen and how its composition affected its physical properties. It was known that bitumens from different crudes had different properties under working conditions, but very little was known as to what gave those particular properties, and he thought that was a subject which might very well be discussed by the Group. The question of the effect that various elements, such as the percentage of sulphur or the amount of wax present in the bitumen, had on the working properties of the bitumen might also be discussed. Very little had been done in that respect, and it was a problem that should be solved if the Group was to fulfil a useful purpose for the users of bitumen generally.

MR. L. J. CHALK said there was one question he would like to ask the author. A photograph of an internal pipe coating with a mirror surface inside had been shown by the author, and when he referred to it he mentioned the term "hydrocarbon." Was there any ulterior motive for that term? Was the coating in fact a blown asphalt, or was it perhaps a coal-tar pitch?

MR. E. A. EVANS said he would like to emphasize what Mr. McConnell Sanders had said about the Group and its membership. He had been a little perturbed when he heard Mr. Jackson say that the membership was going to be confined to members of the Institute, as he was inclined to think that the Institute would lose something if there was that complete exclusiveness.

MR. FUKUZAWA asked the author what was the composition of the asphaltic concrete used in the water-proofing of the Ghrib Dam in Algeria.

MR. J. S. JACKSON, in replying to the discussion, said that the composition of the asphaltic concrete aggregate used in the waterproofing of the Ghrib Dam in Algeria was given in the paper as 10 per cent. filler, 20 per cent. fine sand, and graded limestone chippings, and the composition of the mix was 92 per cent. aggregate and 8 per cent. asphaltic bitumen, 20/30 penetration, but the most important consideration was the amount and nature of the tack coat which established the bond between the asphaltic concrete and the concrete underneath. Variations in the asphaltic concrete itself were not nearly as important as an excess or a deficiency of the tack coat.

With regard to the hydrocarbon lining mentioned, to the best of his knowledge it was an ordinary asphaltic bitumen composition, and he had used the term "hydrocarbon" because it was the commercial term normally used.

With reference to Mr. Evans' remarks, it was hoped that a sufficient support would be forthcoming from members of the Institute to form a virile Group. The meetings of the Group would be open to guests, who could attend them just as they could attend the ordinary meetings of the Institute.

As to the precise composition of bitumen, he hoped that papers would be forthcoming which would deal with the structure and constitution of asphaltic bitumen.

Mr. Broome's remarks were very interesting and informative. In his own reference to the effect of the filler he had emphasized the striking effect of the fibrous nature of the filler, but he agreed with Mr. Broome that the effective fineness of the filler was of fundamental importance.

With regard to jointing compositions, he thought he could say definitely that for hydraulic work they were entirely successful. A little extrusion of the jointing composition did not matter in many hydraulic constructions. In road work any

jointing compositions extruded might be removed by the traffic. The two problems were quite distinct. As long as the jointing remained ductile and adhered firmly it would probably be successful in hydraulic work, and he thought the test that he had described would indicate whether satisfactory adhesion could be obtained under conditions of expansion and contraction.

THE PRESIDENT, in proposing a hearty vote of thanks to Mr. Jackson for his excellent paper, congratulated him on the inauguration of the Group, and expressed a hope that it would meet with great success.

The motion was carried with acclamation, and the meeting then terminated.

MR. L. J. CHALK subsequently wrote: I was particularly interested in Mr. Jackson's remarks on the methods of testing pipe-coating materials, as a number of tests employed in the Geochemical Laboratories are somewhat similar to those described. Our practice in the flow and shatter tests is to employ coatings $\frac{1}{8}$ in. in thickness, whereas Mr. Jackson uses 5 mm. or about $\frac{1}{4}$ in., which I take to be the normal thickness of application of the type of compound to which he refers. In the flow tests we prefer to place the plate in a vertical position in an oven at 60° C. (for compounds destined for use in temperate climates), whereas in the method described in the paper, the plate is supported at an angle of 45° in an oven at 70° C.

Useful data regarding the brittleness of pipe-coating compounds can be obtained by means of the Fraass apparatus. The usual method of preparing the test specimen is not, however, very satisfactory, since it is extremely difficult to eliminate air bubbles and at the same time avoid overheating. Better results are obtained by placing the strip on a hot plate and rolling out the film with a small amalgamated copper roller until the weight of coating on the strip corresponds to the calculated amount. If desired two collars may be placed on the roller to give the requisite clearance between the roller and the strip.

When the pipes are to be buried in a corrosive soil or used to convey corrosive fluids, it is useful to determine the resistivity of the coating to certain acids and electrolytes. For this purpose a film 0.5 mm. thick is prepared, either in the manner indicated above or by using a warm press, and sealed over the end of a glass tube about 4 in. long by 1 in. in diameter. The latter is then filled with the acid or electrolyte and placed in a beaker containing the same medium. An electrode is placed in each compartment and connected by a milliammeter. In due course failure of the coating is indicated by the passage of current.

Passing to the use of micro-asbestos as a filler, I should not agree that its incorporation in pipe-coating materials is a necessity. Admittedly, it is an excellent filler for the purpose, but it is by no means essential and excellent pipe-coating compounds are on the market which have given entire satisfaction in service which do not contain it. Similarly, a wide variety of fillers may be used for joint-filling compounds, and economic considerations do not always admit of the use of the relatively expensive asbestos fillers.

The formulation of jointing compounds for reservoirs demands very careful consideration having regard to the necessity for ensuring water-tightness of the structure. Gradual loss of jointing compound, either by flow or extrusion, must, of course, be avoided. In the case of reservoirs, the width of the joint varies considerably according to the amount of water in the reservoir, and conditions are obviously very much more difficult than in the case of a joint in a concrete road. Adhesion to the concrete is particularly important, but a high ductility is probably not so essential since the rate of movement is extremely slow. The compound should, of course, possess an adequate resistance to flow at all temperatures likely to be encountered in practice, and at the same time should possess a low viscosity at the temperature of pouring in order to enable narrow joints to be easily filled. In many respects, highly plastic bitumens containing no mineral filler are preferable to filled compounds for this particular purpose.

MR. J. S. JACKSON, in reply to the above, wrote: I thank Mr. Chalk for putting forward the descriptions of the tests used in his laboratory. They are distinctly

interesting, and I hope that other people will come forward with descriptions of any tests of interest.

I agree that the use of micro-asbestos as a filler is not essential, and I agree that excellent pipe-coating compounds are on the market in which other fillers have been used with complete success.

In connection with the formulation of jointing compounds for reservoirs, it is difficult, if not impossible, to prevent some measure of extrusion, but this should not have any detrimental effect upon the joint provided the composition used has a satisfactory ductility and satisfactory adhesive properties as judged by the test described in my paper.

A VISIT TO RUSSIAN OIL DISTRICTS.*

By H. G. KUGLER, Ph.D. (Member).

INTRODUCTION.

OF the long excursions that took place after the final sessions of the 17th International Geological Congress at Moscow, the one to the Uralian and Caucasian oilfields will remain in the memories of the participants, who represented about fourteen different nations.

The five well-illustrated official guide booklets prepared under the direction of A. J. Krems contain all essential geologic information on the areas visited, especially when studied in conjunction with the guide-books for the Caucasian and Permian excursions.

Furthermore, a comprehensive account of this visit, with valuable details on oil-engineering questions, was recently published by L. D. Wosk.¹⁵ It is proposed, therefore, to confine this paper largely to the description of some oilfields and of a few features that have hitherto received only cursory recognition.

The map of the Apsheron Peninsula has been taken from Goubkin's "Tectonics of the South-eastern Caucasus,"³ with certain modifications by the author. The remaining figures have been re-drawn from the guide booklets.

The author is indebted to the Directors of the Central Mining & Investment Corporation, Ltd., for their liberality in affording him the opportunity of taking part in this efficiently organized excursion to regions abounding in interest both geologically and technologically.

Thanks are also due to the Russian colleagues for the enthusiasm shown and help offered during the entire Congress and excursion.

OIL DISTRICTS TO THE WEST OF THE URALS.

General Geographic Remarks.

The first destination of the excursion was the Permian district. As long as one hundred years ago Murchison attributed the sediments of this area to the Permian system. During the eighteenth century the exploitation of cupriferous sandstones of Upper Permian age, the more recent discoveries of deposits of potash salts, and finally the finding of commercial accumulation of oil maintained the continuous attention of mining engineers and geologists.

From geologic and morphologic view-points the area between Moscow and the Central Ural mountains can be subdivided into :—

1. The Russian Platform.
2. The Preduralie.
3. The West Ural.

* Read to a meeting of the Trinidad Branch on June 22nd, 1938.

1. *The Russian Platform* represents a typical peneplain of horizontally-bedded Palæozoic, Mesozoic and Kainozoic sediments. The river system is senile, exhibiting little signs of erosion.

Although regional folding is absent, signs of post-Palæozoic disturbances are present. At various periods the pre-Cambrian basement rocks have been fractured. Along the fault lines, blocks moved vertically and horizontally, disrupting the superimposed younger beds. Flexured faults or long, narrow anticlines resulted, such as, for example, that of the "Samara Bend," with its oil pools in Palæozoic rocks. The oldest beds outcropping on the Russian Platform are of Carboniferous age. Devonian has been found only in bore-holes. The Upper Permian formation has the widest distribution. Marine Triassic is missing; in its place, the Upper Permian sediments gradually change to terrestrial deposits carrying dinosaur remains (Vestlugian).

2. *The Preduralie* is closely related to the Russian Platform. It differs, however, in that it possesses scarcely discernible folds of great length and width. Farther to the east, blocks lifted to heights of 400 and 500 m. are an additional cause for the considerable rejuvenation of erosion. The axes of the folds are parallel to the main strike of the Ural Range—namely, north-south. Some of these regional uplifts are complicated by faults, and even by thrusts. The presence of reef limestones in Lower Permian and Upper Carboniferous are characteristic of the Preduralie.

3. *The West Ural* forms a part of the extensive Uralian geo-syncline. Although the Central Ural, with its Variscan orogeny, consists of intensively folded and overthrust rocks, the West Ural is considerably less disturbed in so far as Devonian and younger beds are concerned.

The boundary between Preduralie and West Ural has been drawn along the north-south directed depression formed by the soft Artinskian sediments. The West Ural is a mountainous country with beautiful valleys containing swift-running rivers, and even rapids. The hilltops, which are the remains of the erosional surface of a former peneplain, reach heights of 600 and 700 m. They are often gently undulating with marshy watersheds, and are covered by forests.

Geologic History.

In order to arrive at a reasonably clear conception of the nature of occurrence of oil to the west of the Ural Range, a short synopsis of the geologic history is necessary. Since a correct interpretation of the palæogeographic conditions is bound to lead to discoveries of new oilfields, the Russian geologists attach great importance to detailed biostratigraphic studies.

During Lower Palæozoic time the Russian Platform was a land-mass. The area of the present Ural Range was, however, covered by sea. Although little is known of the extent of this sea, lagoon and shoreline conditions are exemplified by conglomerates, sandstones, thin bedded limestones and reef limestones. Upper Cambrian deposits are missing. Ordovician sandstones and conglomerates are resting directly on Middle Cambrian beds, as in the South Urals. Lower Devonian sediments show a distribution similar to those of the Cambrian.

During Middle Devonian time an important marine transgression took

Table of the Palæozoic.

Permian	{ Tartarian. Kazanian- Zechstein Kungurian Artinskian. }	} Ufimian.
Carboniferous	{ Uralian. Moscovian Dinantian }	{ Samarian. Moscovian S. str. Martyanian. Visean. Tournaisian. }
Devonian	{ Famennian Frasnian Givetian Eifelian Coblentzian Gedinnian }	{ Limestone with <i>Spirifer tornacensis</i> . Bituminous limestone with <i>Atrypa</i> and <i>Manticoceras</i> <i>intumescens</i> . Limestones with <i>Stringocephalus burtini</i> . Limestones with <i>Conchidium baschkiricus</i> . Sandstones, shales, and subordinate limestone. }
Silurian	{ Gothlandian Ordovician Potsdamian }	{ Continental and marine deposits. Shales, sandstones, and tufa with diabase. Corals. (Apparently absent.) }
Cambrian	{ Acadian Georgian. }	{ Dolomites, quartzites and phyllites with <i>archæocyatus</i> . }

place. Coming from the east, the sea flooded the Russian Platform and extended far into Poland. Parts of the West Ural still formed islands. Between these islands and farther to the west, bituminous limestone and shales came to rest beside sands and conglomerates.

During Upper Devonian time the submergence reached its maximum, with the resultant deposition of well-bedded, dark, pyritic limestone, bituminous shales, dolomites and crystalline limestones. These Middle and Upper Devonian bituminous beds are some of the main source-rocks of oil to the west of the Urals.

At the end of the Devonian, the north-west part of the Russian Platform emerged, with the consequent retreat of the sea eastwards. This recession continued throughout the Lower Carboniferous (Tournaisian), and the coal basins of Moscow and Kizel were formed. However, after the deposition of the Lower Visean coal-beds, regional subsidence again set in, and a thick series of Middle and Upper Visean *gigantella* limestone was deposited intercalated in places with bituminous shales.

Middle and Upper Carboniferous as well as Permian times were characterized by the Variscan orogeny, and this, in turn, was responsible for the creation of the Ural Range. Folding and contemporaneous erosion started to the east of the Urals. The clastic material was transported to the west, and the rapid lateral changes of the Middle Carboniferous sediments bear witness to the littoral conditions prevailing during this time.

The Visean limestones were succeeded by lagoonal deposits in the form of dolomites covered by sands and clays. Orogenic movements gradually affected more westerly situated regions. There are no marine Upper Carboniferous or Permian sediments to be found in the Eastern Urals, but plant-bearing Upper Permian was discovered in a few remains protected by dislocations.

During the rising of the Central Ural, the area of the present West Ural was continuously covered by a sea which produced successively the Upper

Visean limestone and a thick series of Moscovian and Uralian Fusulina limestone. Similar conditions persisted in Lower Permian times, and reefs of Schwagerina limestone fringed the most westerly deposits of clastic material washed from the advancing chains. Increasing quantities of sands, clays and marly limestones were deposited between the reef limestones and the mainland of the Central Ural. These littoral deposits are grouped under the stratigraphic term "Artinskian," and are several thousands of metres in thickness. In its nature, the Artinskian can be compared with the pre-Alpine Molasse.

The main phase of Variscan movements affected the Central Ural during the Lower Permian, and the Artinskian sediments increased to a belt of 80 km. in width, whereby the conglomerates came to rest closer to the main Ural Range.

The Kungurian stage of the Permian represented a time of general emergence of the Western Ural and a retreat of the sea westwards. Numerous lagoons and salt marshes were in existence, and potash salts, gypsum and dolomites became interbedded with red clays and sands. The regional emergence went steadily on, so that in Upper Permian time the entire West Ural and Preduralie was covered by a flat, monotonous desert with large deltaic fans of debris. The sediments of this period are generally known as Ufimian rocks. West of the desert extended a wide basin alternately flooded by brackish and marine water, which produced the sediments known as Kazanian. The marine deposits are closely related to the German Zechstein. There is a distinct similarity between the palæogeographic conditions of this Upper Permian time and those attaining to the east of the Caspian Sea at the present time.

Towards the end of the Permian (Tartarian) the Kazanian Sea had dried up. The last remains of fresh and saline marshes were covered by red terrestrial sediments of Ufimian aspect.

The remarkable similarity of the Permian sediments of the Western Ural to those of New Mexico and West Texas was a source of great interest to the American geologists.

In Mesozoic times the Russian Platform, the Urals and the Donetz basin were a land-mass over which dinosaurs roamed. However, during Lower Jurassic time subsidence set in to the south, and the sea transgressed the Donetz basin. The present Volga Valley up to Samara was subsequently flooded by the Middle Jurassic sea. Finally the Thetis sea of the Upper Jurassic transgressed the Preduralie depression and joined the Northern Ocean. To the east the Urals, and to the west the Russian Platform, remained land. The influence of the Northern Ocean prevented the growth of limestone. Only in the Donetz basin and in the Orenburg region are thin layers of lime found. In the Volga region oil shales were deposited, and are exploited near Kashpur

The Cretaceous and younger rocks do not contain important oil deposits; hence their history is not commented on in this paper.

Oilfields.

Amongst the oilfield districts to the west of the Urals, the excursionists visited some of the Permian and Bashkirian fields of the Preduralie and the Kuibishev district of the Russian Platform. These fields produce their

oil almost exclusively from Carboniferous and Permian limestones and dolomites. In Syzran thin layers of Carboniferous sandstone must be added to the list of reservoir rocks.

1. *Permian Region*.—In 1929 the first commercial oil was obtained from the Gorodki structure situated about 65 km. north-east of the town of Perm. It was not, however, until 1934–1936, when the development of the Krasnokamsk district supplied additional information as to the nature of the reservoir rocks and the mode of occurrence of oil, that Gorodki and other structures received more attention.

Geophysical prospecting greatly aided in the exploration of the irregularly undulating limestone structures concealed below younger beds. At the end of 1937, about fifteen structures were known. The main Prekamie structure of about 75 km. length and 16 km. width reveals a number of so-called “brachy-anticlinal uplifts.” It is, however, a moot point whether these oil-traps are really tectonic features. They may well be reefs which grew above the levels of the normally deposited limestone of Uralian age, in which case it is assumed that the Kungurian and Kazanian anhydrites, dolomites, reddish clays and sandstones imitate structural conditions solely by compaction of the compressible deposits superimposed on the slopes of the reef limestone.

In *Krasnokamsk Field* the first oil was encountered at the shallow depth of 160–180 m. in Kungurian dolomites. Better concentration was discovered in the limestones and shales of the Martyanian stage of the Carboniferous limestone and at depths between 900 and 950 m., and there is every reason to expect additional accumulation in the Viséan and Tournaisian stages of the Lower Carboniferous as well as in the Devonian.

The occurrence of dolomitic layers within the limestone is a subject of considerable argument amongst Russian geologists. Some are inclined to consider them to be proof of surface exposure and weathering, whilst others believe them to be a part of the normal sedimentary conditions in growing reefs. It is quite obvious that reefs can become dolomitized in a “status nascendi”; moreover, they can be full of holes, and along their outer rims can be covered with reef debris merging into normal shaley sediments without having been exposed to weathering. A different matter is the explanation of the presence of oil in these reef limestones. Apart from a possible vertical migration of oil from known deeper source-rocks, one has to consider the possibility of deposition of source-rocks in the stagnant depressions between growing reefs or between reefs and the old land-mass, since such sediments have commonly been known to form black muds with a considerable content of bitumen.

The main oil-bearing bed of the Martyanian limestone attains a thickness of 40 m. in which the volume of oil represents about 20 per cent. of the rock volume. The oil of the Kungurian dolomites has a specific gravity of 0.925 with 22 per cent. light fractions between 100° and 270° C. The oil of the Martyanian shaley limestone has a specific gravity of 0.846 with 36 per cent. light fractions. Very little gas is associated with the oil, hence the flowing life of the wells is short. The wells are spaced at approximately 250 m. centres.

2. *The Bashkirian Region*.—On the way from Ufa (the capital of the



PLATE 1.

[Photo: E. Lehner]

TRATAU, "SHIKAN," REEF OF PERMIAN LIMESTONE.



PLATE 2.

[Photo: E. Lehner]

NORTH KABRISTAN, VARIEGATED APTIAN SHALES POSSIBLY PPLICATED THROUGH
GLIDING.

[To face p. 72.]



[Photo: H. G. Kugler]

PLATE 3.

ELDAMA DOME, DAGHESTAN. AVALANCHE-LIKE SLIDE MASS OF UPPER
CRETACEOUS MARLSTONE IN EOCENE SHALES.



[Photo: E. Lehner]

PLATE 4.

ATLY BOYUN, DAGHESTAN. "NEPTUNIAN DYKES" IN MAIKOP SHALES.

Bashkirian Republic) to the oilfield of Ishimbaevo, one is continuously struck by the occurrence of abruptly rising beehive and whaleback-shaped limestone hills extending along the east side of the Belaya River Valley. "Shikan" is the local name of such a monadnock, amongst which the Tra-Tau imitates an almost perfect volcanic cone rising about 280 m. above the alluvial flats (Plate No. 1). These white limestone masses contrast strongly with the gently rolling landscape of the surrounding Upper Permian red beds. D. V. Nalivkin and others recognized the reef character of these "Shikans," although his explanation has not received general recognition amongst Russian geologists. The abundant occurrence of Schwagerina princeps would place these limestones to the Uralian. The palæontologic studies of Guerassimov and others lead them to consider the rocks of the Shikans to be of Lower Permian age, and, what is more, to be a facies of the Artinskian. No doubt the Uralian facies, with its reciprocal fauna, persisted into the Lower Permian. Similar conditions are known from the Silurian coral reefs of Gotland, the algal reefs of the Dolomites of Southern Tyrol and the sponge reefs of the Upper Jurassic of Swabia, to mention but a few different reef-formers exhibiting upward growth with a lateral facies carrying different faunas. In other words, the Shikans represent a facies clearly restricted laterally as well as vertically. The pure limestone contains less than 1 per cent. of insoluble material. The slopes of the reefs change laterally to debris and muddy limestone. Orogenetic movements may have slightly affected the limestones, and are very likely responsible for the semi-diapiric folds in the superimposed Kungurian anhydrites, gypsum and shales (see section through Ishimbaevo field). Ufimian red beds of an almost horizontal position cover the older formations.

The Permo-Carboniferous reefs of the Bashkirian region lie parallel to the Ural Range and extend in a northerly direction, probably into the Permian region. They disappear towards the west of Ishimbaevo, and likewise to the east of the Tra-Tau, being thus confined to a belt of about 20 km. in width.

The *Ishimbaevo Field* is associated with a buried "shikan," and thus serves as a typical representative of about thirty-five others at present tested or under exploration. As far back as 1768 oil was reported to seep from Kungurian beds outcropping in the centre of the present Ishimbaevo field. Some prospect drilling was carried out in earlier years, but it was not until 1932 that the first commercial wells were brought in. During the course of time, three different dome-like uplifts have been delimited by drilling, and a proven area of about 5 sq. km. has been marked out. The oil is present only in the cavities of the dense limestones of the higher parts of the reefs.

The thickness of the producing horizon of Ishimbaevo is between 50 and 300 m. Production is found to a depth of about 1000 m. Bottom- and edge-water surround the oil pools. The largest initial production was 800 tons per day from one well. The average initial production is about 100 tons of an oil showing a specific gravity of 0.878 with 44 per cent. light fractions and a sulphur content of 2.5 per cent. The average well spacing is about 100 m. (330 ft.) and even less. It is claimed that no interference exists between wells. All wells are acidized and respond

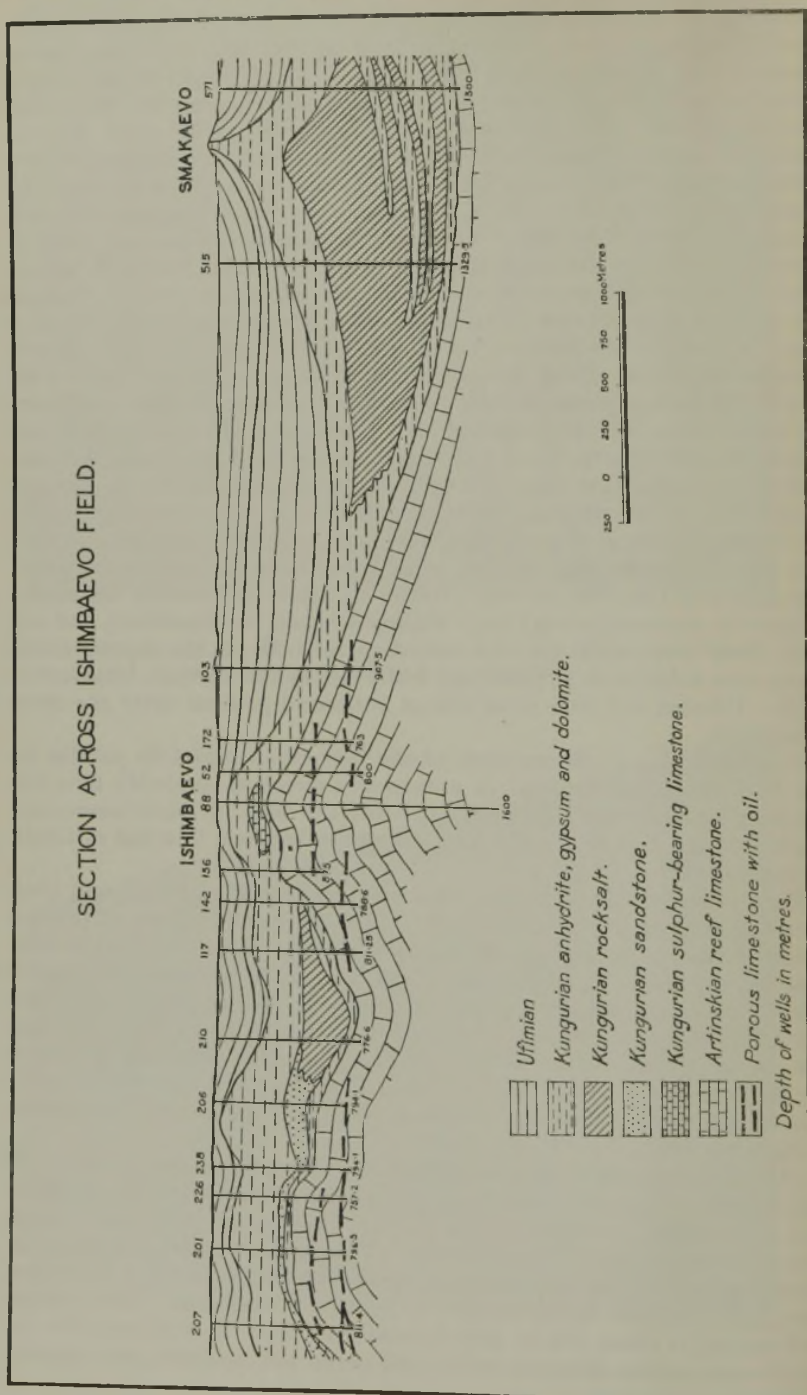


FIG. 1.

initially, but fall off fairly rapidly. The close spacing of wells is, indeed, a strange feature for limestone fields, and only tends to prove the existence of oil in cavernous horizons which are the product of normal sedimentation and not of secondary fracturing; otherwise communication over a greater distance would result.

At *Smakaevo*, a pseudo-diapyrlic fold occurs about 8 km. east of Ishimbaevo (see Fig. 1). Kungurian gypsum with anhydrite pierces through Ufimian beds. The underlying limestone is completely despatched from this shear fold. It actually forms a depression filled with anhydrite. Such incipient diapirs are common in the Fars of the Iranian oilfields, and according to G. M. Lees, are termed "scruff-folds" by the geologists of the Anglo-Iranian Oil Co. To what extent hydration of the anhydrite plays a rôle in bringing about such structures is not clear.

3. *The Kuibishev Region*.—The route from the Bashkirian oilfields to the old town of Samara (now Kuibishev) on the Volga passes through a rolling landscape of Permian red beds. It appears that, wherever the Carboniferous has been warped or faulted underground, there are prospects for oil. A few derricks spotted from the train seem to support this assumption.

At Kuibishev, the Volga forms a hairpin bend of 200 km. in length.

This so-called Samara bend or "Samarskaya Luka" is undoubtedly connected with the uplift of the Jiguli hills, one of the flexures in the Russian Platform mentioned above. The outcrops of Permian and Upper Carboniferous limestones have always been famous for their richness in fossils. For this reason the Jiguli hills form a classical locality for the study of the Upper Palæozoic of the Russian Platform. Deep drilling disclosed Middle and Lower Carboniferous as well as Devonian.

The latter is mainly composed of greenish-grey marls intercalated with limestones of possibly 210 m. total thickness. These Devonian shales can be correlated with the equivalent oil shales of the West Ural. The Carboniferous attains a thickness of about 1000 m. of limestones, with subordinate marls and clays. Directly overlying the Schwagerina limestones of the top Carboniferous is a series of dolomites and gypsum belonging to the Lower Permian, which have a total thickness of about 70 m.

The overlying Kazanian is about 110 m. in thickness, and comprises a series of dolomites, gypsum and clays; it is sub-divided into a lower Spirifer and an upper Conchifer zone. The Kazanian represents a recurring depositional cycle of dolomite layers rich in fossils and gypsiferous beds poor in fossils, with a gradual tendency to decrease in the average fossil content upwards.

The successively younger Tartarian consists mainly of red clays and marls with sandstones. Mesozoic beds with well-developed fossiliferous marine Jurassic and Cretaceous beds overlap the older formations.

The Jiguli hills form moderately undulating uplifts limited to the north by an east-west striking fault or flexure covered by the Volga River alluvials. Thus the north flank of the Jiguli structure shows dips of 40–45°, whereas those of the south flank do not exceed 3°. The Jiguli disturbance is of Lower Pliocene age, whereas the subsidiary uplifts are pre-Jurassic.

Exploitation of asphalt from Upper Carboniferous Schwagerina lime-

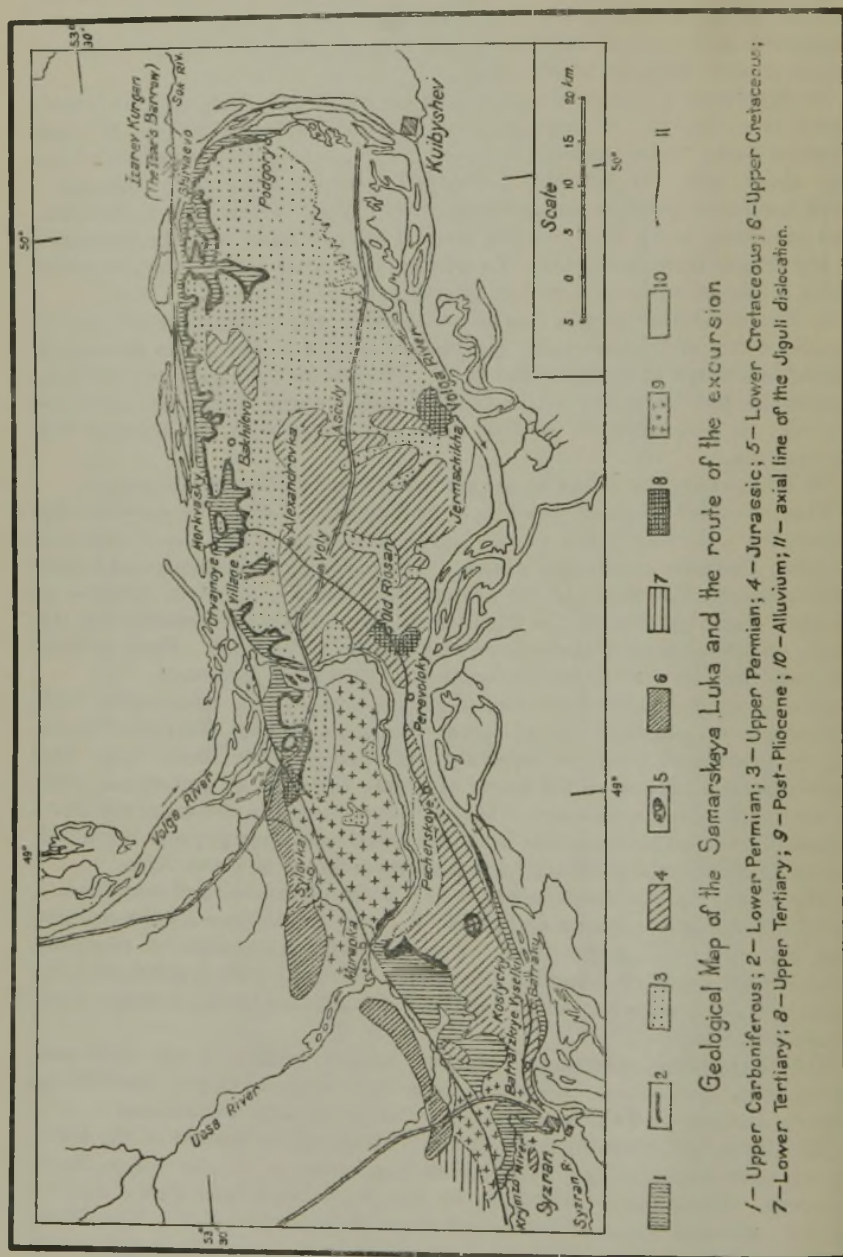


FIG. 2.

stone and Permian dolomites has been carried out for a long time. The exploited beds are between 80 and 100 m. in thickness. Sands of the transgressive Jurassic are likewise mined. The presence of asphaltic limestone pebbles in Bathonian sands is a proof of the dissemination of oil prior to the deposition of Jurassic sediments. The source of this oil is undoubtedly in the Middle and Lower Carboniferous.

The *Syzran Oilfield* supplied the first commercial production from Carboniferous beds in Russia. This field is associated with a very gentle uplift along the western prolongation of the Jiguli dislocation. An upper, poorly producing, shaley limestone horizon lies in the Martyanian, and is equivalent to the main producer of Krasnokamsk. The main producing horizon of Syzran occurs at a depth of 1000 m. in thin Tournaisian sands on top of a massive limestone which is barren of oil. The productive area of the Syzran pool is not yet delineated, but could scarcely be more than 2500 acres. Future oilfields will undoubtedly be discovered along the Jiguli hills, their eastern and western prolongation.

THE CAUCASIAN OIL DISTRICTS.

General Remarks.

Russia's leading position in the world's oil industry at the end of the last century was due to the Caucasian oilfields. Even at present the bulk of U.S.S.R.'s production is derived from the old fields and their lateral extensions as developed subsequently. Apart from oil, the Caucasus is richly mineralized, although the statement of one of the Russian interpreters that "the Caucasus lacks in nothing but bird's milk" is somewhat exaggerated. It may, however, be almost true when applied to the resources of the entire U.S.S.R.

A very condensed synopsis of the major geological features of the Caucasus will assist in the conception of the vastly different types of structures found in the Circumcaucasian oilfields.

On both sides of the main range of the Caucasus are located two rigid plates: to the north the Stavropol mass between the Kuban and Terek valleys, and to the south the Transcaucasian plate, divided by the saddle of Suram into a Rion and Kura segment.

These resistant masses are composed mainly of strongly metamorphosed and dislocated pre-Cambrian rocks. Parts of these basement rocks are found to compose the old backbone of the main Caucasus range. Besides pre-Cambrian schists and granites, this backbone also consists of Cambrian, Silurian, Devonian, marine Lower Carboniferous, continental Middle and Upper Carboniferous, terrestrial Lower Permian, marine Upper Permian and Triassic. All the sediments of these periods have been over-stepped by the Lower Jurassic, which forms the base of an enormous thickness of Mesozoic deposits. The geosyncline responsible for these Mesozoic sediments extended between the Stavropol and Transcaucasian plates and formed a part of the Thetis Sea in which thousands of metres of Jurassic and Cretaceous beds were deposited. To the south they are generally found in a flysch-like facies, whereas to the north mainly open sea deposits came to rest. During Tertiary time the two plates moved together as a result of Alpine orogeny, which led to the creation of the

mighty Caucasus Range with its crowning volcanoes along the main lines of disturbance.

The fan-like arrangement of folds is also a result of this orogeny of the Central Caucasus. A part of the pressure was released in tangential movements, resulting in overthrusts towards the south. The north side is characterized by Mesozoic sediments dipping comparatively regularly to the north. The eastern segment of the north flank is, however, strongly disturbed. Cretaceous is there found to be thrust over Tertiary. The south-east and north-west plunging ends of the Caucasus reveal still more intensively folded Mesozoic and Tertiary beds. Towards the Apsheron Peninsula they reach a total thickness of about 12,000 m. : Pleistocene \pm 200, Tertiary \pm 4500, Cretaceous \pm 3000, Upper and Middle Jurassic \pm 2000, and Lower Jurassic \pm 2000 metres.

Apsheron and Kabristan Region.

The main oilfields of the Caucasus are associated with its south-eastward plunging end. Although the stratigraphy of this region has been studied for a long time, divided opinions still exist as to the actual age of some of the formations. The stratigraphic section that follows has been published by Goubkin,³ and has been amended by the author in accordance with the latest information.

Structural Conditions.

The most striking structural features of the Apsheron Peninsula are the "pseudo-diapiric" uplifts with their crescent and hook-like trends of folding (see map opposite, Fig. 3). These structures and their relation to the occurrence of mud volcanoes, as well as their genetic association with an area of almost continuous subsidence, have been dealt with in a most interesting paper by I. M. Goubkin.⁴ The true value of crescent-like folds still awaits a satisfactory explanation. W. Staub¹² uses the term "Rahmen Faltung"—i.e., "frame-folding"—for this ring-like structural pattern of Apsheron. He suggests lateral resistance to otherwise unrestricted folding as the main cause for the occurrence of arched segments along axial regions. Similarly, cross and ringfolds, so Staub contends, would occur only where there is hindrance to a free development of anticlines.

As far as Kabristan and Apsheron are concerned, gravimetric surveys clearly demonstrate the presence of a tectonic depression to the east of the plunging Caucasus. This north-south directed depression must have already existed in late Tertiary time, as is manifested by the changes in thickness of the Producing Series—namely, zero in the Sumgait Valley to the west, and about 2000 metres on Artem Island to the east. The main line of Caucasian folding is still preserved in the Cretaceous and Eocene structures of North Kabristan, of which the important Kown anticline should be specifically mentioned. The strong south-eastern plunge of the Caucasus is reflected in the long anticlines of South Kabristan and the extraordinary meridionally directed segments of some Apsheron uplifts.

Pseudo-diapiric Folds.—L. Mrazec, who first introduced the term

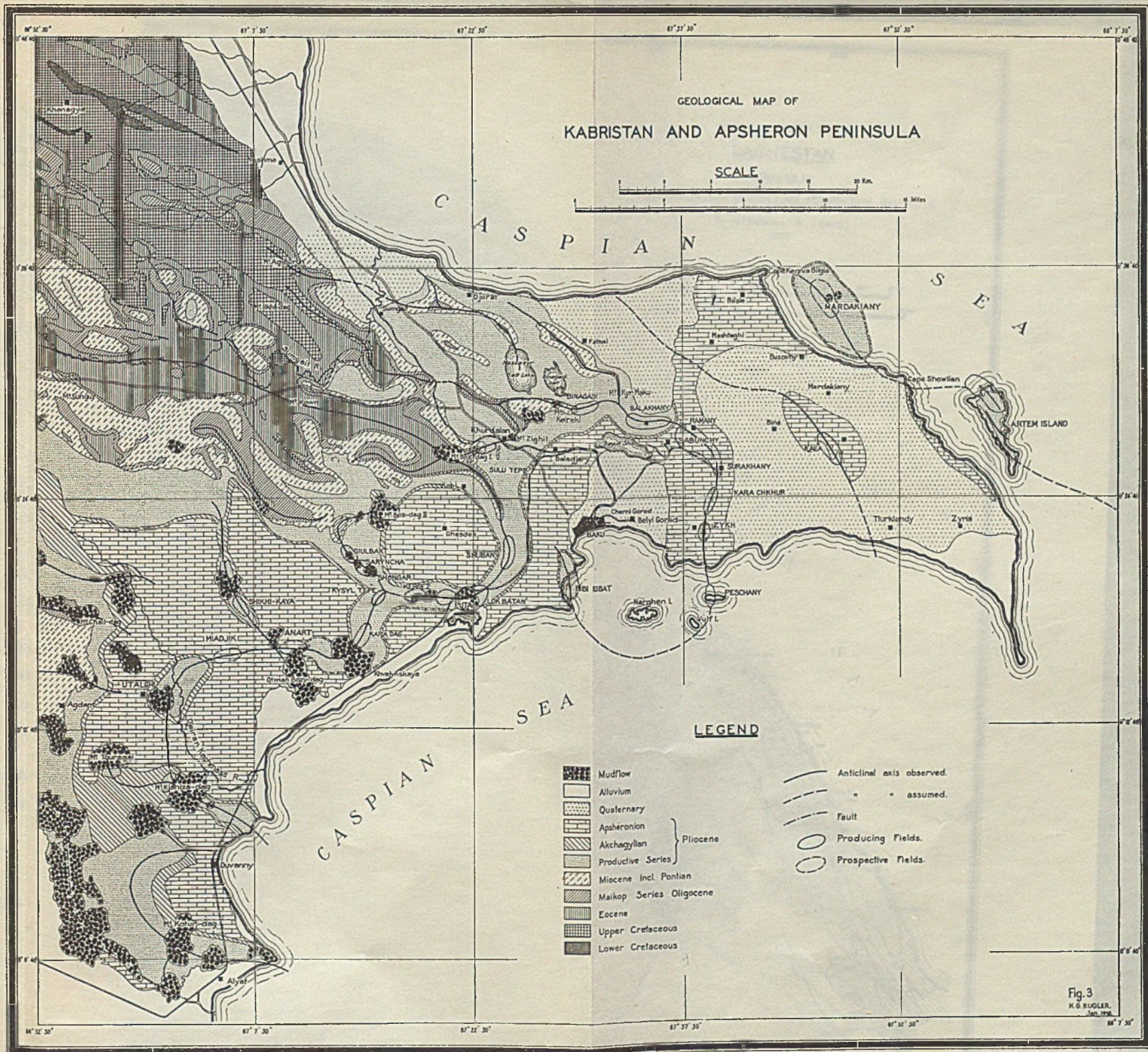


FIG. 3.

Generalized Section of South-Eastern Caucasus.

		Lithologic Characteristics.	Thick- ness in metres.
Quaternary	Recent. Ancient Caspian deposits.	Sand and clay predominate. Conglomerate sand, shelly lime- stone, etc.	± 10 126
Upper Pliocene	Apsheonian. Akchaghylian. Producing Series.	Upper 350 m. sandy limestone, clay and sand below.	720
		Clay, sand and volcanic ash. Sand, silt and clay.	± 60 ± 1500
Unconformity extensive			
Middle Pliocene	Pontian.	Dark grey clay, siliceous marl.	320
Unconformity in Kabristan			
Upper Miocene.	Maeotic-Upper Sarmation.	Thin-bedded clay and diatoma- ceous earth.	136
	Middle Sarmation.	Clay with siliceous marl.	20
	Lower Sarmation.	" " "	250
		Spaniodontella-Karagan horizon.	?
Middle Miocene	Second Mediterra- nean Stage	Spiralis beds.	65
		Chokrak	500
Lower Miocene- Oligocene	Maikop Series.	Laminated clays, partly bitu- minous.	330
Eocene	Kówn Series.	Shale, marls, sandstones.	600
Palæocene	Sumgait Series.	Clay, marls with glauconitic sandstone.	100
Upper Cretaceous	Ilkhidag Series.	Shales, coarse sandstone, marl, etc.	300
	Yunusdag Series.	Pink, fucoidal clay, glauconitic sandstone.	200
	Kemchi Series.	White marls, breccia limestone, sand.	250
	Sub-Kemchi Series.	Shales, marls, block conglomer- ates at base.	450
Lower Cretaceous	Aptian.	Pink and grey marly clay, sand- stone and conglomerates.	500
	Barremian.	Laminated clays and sandstone.	600
	Neocomian.	Calcinated sandstones and lime- stones. Flysch facies.	?
Upper Jurassic	Tithonian.	Massive limestone and dolomite.	?
Middle Jurassic	Khinalug Series.	Shales with plant remains.	1370
Lower Jurassic	Lias.	Black shales with subordinate marly sandstone.	?

“diapyric fold” in geologic literature, defined it as a structure in which the core consisting of older soft material has pierced younger beds. Kraus, Krejci-Graf, Bogdanowitch and de Cizancourt made additional and important contributions to the knowledge of diapyrism by stressing an upward movement of the core region as being independent of the general dip of the flanks of the uplift. The following lines are quoted from Goubkin⁴ in his discussion of diapyric folds :—

“In the process of folding, at the time when deposits had been forming, tangential forces had, apparently, already predetermined the lines along which anticlinal folds are situated. On the one hand, they created weakened points at the arching portions of anticlines and, on the other hand, they caused unevenness of the underlying strata which results, in turn, in the uneven accumulation of deposits. This uneven accumulation of deposits originated zones of high and low pressure which, in turn, led to the further differentiation of the process where the predominating importance was assumed by the force of gravity, the force of vertical pressure acting upon plastic masses and impelling them to move along the lines of least resistance into the zones of low pressure.”

Although one has to agree with the fundamental correctitude of this interpretation for most of the Apsheronian and Kabristan uplifts, it may be judicious to stress the part which uneven accumulation of deposits must have had. Once it is proved that the very reduced thickness of sediments in any crestral region represents the normal equivalent of the mighty deposits on the flanks and synclines, there would be no need for the assumption of considerable vertical movement of the beds. If, however, ordinary compression between competent masses can be considered responsible for the vertical appearance of the cores, the name “*pseudo-diapyric*” is suggested, and would be more applicable to the nature of the Apsheronian folds.

One of the main characteristics of “*pseudo-diapyric*” folds is the occurrence of re-deposited fossils, especially foraminifera, in each younger bed occurring in the crestral region. The author repeatedly questioned geologists of the Baku region concerning the mode of distribution of foraminifera throughout the Tertiary series, but could not obtain a satisfactory answer, as apparently little attention was paid by the exploitation geologists to the study of foraminifera. According to one of the leading geologists, the reason for this disregard of foraminifera was their unreliable vertical distribution. Such a statement, however, indicates the marked difference in conditions from those ascertained by careful investigations of Cretaceous and Tertiary sediments outcropping in the synclines of North Kabristan. Should, therefore, any study of marine sediments in the crestral regions defy stratigraphic sub-division based on foraminiferal research, it would indicate re-deposition of foraminifera within these axial regions. Apart from the importance of arriving at a clearer conception of the nature of the extraordinary occurrence of reduced thicknesses of older sediments along the uplifts of Kabristan and Apsheron, it is possible to eliminate the undesirable elements of re-worked foraminifera and operate with type assemblages only.

Fig. 4 represents the general conditions observed in these pseudo-diapiric structures.

At Zigil Piri the excursionists visited a trench cut through the crestal region of the local uplift. Below the unconformably overlapping Producing Series followed a normal succession of Pontian, Diatomaceous Series, Chokrak Spirialis and Maikop, the total outcropping thickness of which does not exceed 100 m., whilst their full development would be at least 1500 m. Such conditions are not in agreement with true diapirism, wherein the up-squeezed old core is found in juxtaposition with more gently dipping younger beds of normal thickness. Until the true nature of such reduced series is explained, it would be advisable to discard the term "diapiric" for the Kabristan and Apsheron structures and substitute "pseudo-diapiric."

Mud Volcanoes.—Every reservoir of the Cretaceous and Tertiary deposits of the south-eastern end of the Caucasus can be oil- and gas-bearing when in a tectonically favourable position. It is therefore not surprising to find numerous indications of the presence of oil. The most important of

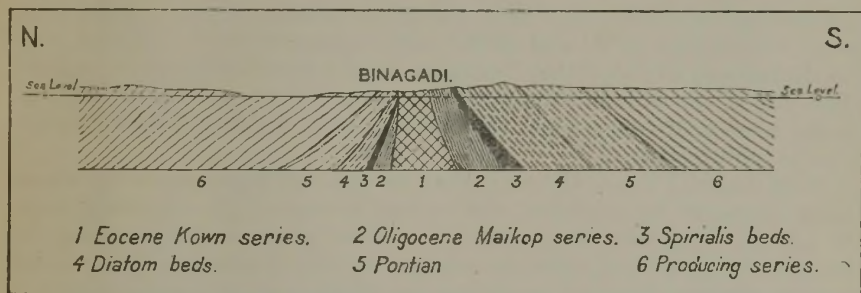


FIG. 4.

these are the mud volcanoes, which deserve special attention on account of their size and number. The cones of the Touragai, Kianzi Dag and Kalmas, to mention but a few, rise to heights of 400 m. above the adjacent Caspian Sea.

The quantity of gas which continuously escapes from these vents must be stupendous. Eruptions are naturally most spectacular and impressive, especially when accompanied by spontaneous ignition and prolonged fires, during which the mudflows can be fused and burnt to porcellanite. In January 1922 the Otman-Boz-Dag exploded. According to S. Kovalevsky, the smoke reached a height of 14 km. In March 1924 the flames of the Touragai could be seen from a distance of 700 km.

Goubkin justly stressed the great importance of the study of the phenomenon of sedimentary volcanism as an aid in elucidating problems concerning the migration of oil and gas. He convincingly demonstrated the genetic association of mud volcanoes with pseudo-diapiric structures. Additional information obtained from Russian geologists during the visit clearly indicated the basic truth of his contention. Mud volcanoes which at one time were considered to be situated along a flank or even in synclines could, on closer investigation, be connected with hidden pseudo-diapirs.

The practical importance of this knowledge is obvious to all who have experienced repeated failures to drill through the squeezed and shattered cores of axial regions underlying gently dipping superficial structures crowned with mud volcanoes. The early history of drilling on the Apsheron and Taman peninsulas is eloquent proof of the difficulties which are caused by such conditions. Similar cases can, no doubt, be quoted from other petroliferous areas distinguished by the presence of mud volcanoes.

Amongst the ejectamenta found in the mud volcanoes of the south-east end of the Caucasus, are components of Tertiary, Cretaceous and even Jurassic rocks. It is obvious that gas alone cannot be responsible for the formation of the clay-breccia associated with these erratic blocks of different formations. Many cases are known where mud breccias have been squeezed like toothpaste through available cracks without the assistance of visible gas or water. Such cracks and fault lines are the normal accompaniment of repeated epianticinal collapse, which, in turn, is often the result of earlier exudations of large masses of mud. Drilling revealed the existence of thick deposits of mudflows in the Producing Series and younger beds. Upon subsequent compaction and tectonic movements, such intraformational mudflows may have started to migrate again. In this way rock components of several cubic yards content have found their way to the surface, although they belong to depths thousands of metres below. The gas of the volcanoes is not necessarily directly related to oil, and many large mud volcanoes to the east and west of the Caspian Sea exude almost pure methane.

Ring-shaped Folds.—Although the crescent and hook-shaped trends of folds appear to be controlled to a certain extent by the competent mass of 350 m. of Apsheronian sandy limestone, their occurrence must be associated also with the afore-mentioned tectonic depression still occupied by the Caspian Sea. At least 4500 m. of Tertiary sediments alone are estimated to underly the Apsheron Peninsula. The slope of the base of the Producing Series must have been at least $2\frac{1}{2}$ degrees. If one considers an additional sinking of the shelf, coupled with a rising of the "hinterland," one is justified in assuming a gradual sliding of the great masses of soft Tertiary sediments. In this respect the Maikop clays and the Diatomaceous beds make admirable lubricants for any such movement. Incipient folds of Cretaceous and Eocene rocks initially aligned in conformity with the main Caucasus direction were bent or over-ridden.

Many indications of sub-aqueous solifluction could be noticed in the Caucasian region. Those of Daghestan will be discussed later. One of the most impressive features was seen on the way from Kiliazy to Alti Agach (see Plate No. 2). Intensely folded Aptian pink-and-white-coloured marly shales were found to be overlain by gently dipping sandstones and shales of Subkemchi beds. One could but have the impression of a tremendous flow of these Aptian shales prior to the deposition of the Subkemchi beds with their basal block conglomerates, especially in view of the fact that more rigid beds below these shales showed no signs of such intense plication.

The detachment and individual gliding of any of the more rigid formations on lubricating shales is conceivable. Finally, the entire Cretaceous series may have sheared from the massive Tithonian limestone, whereby

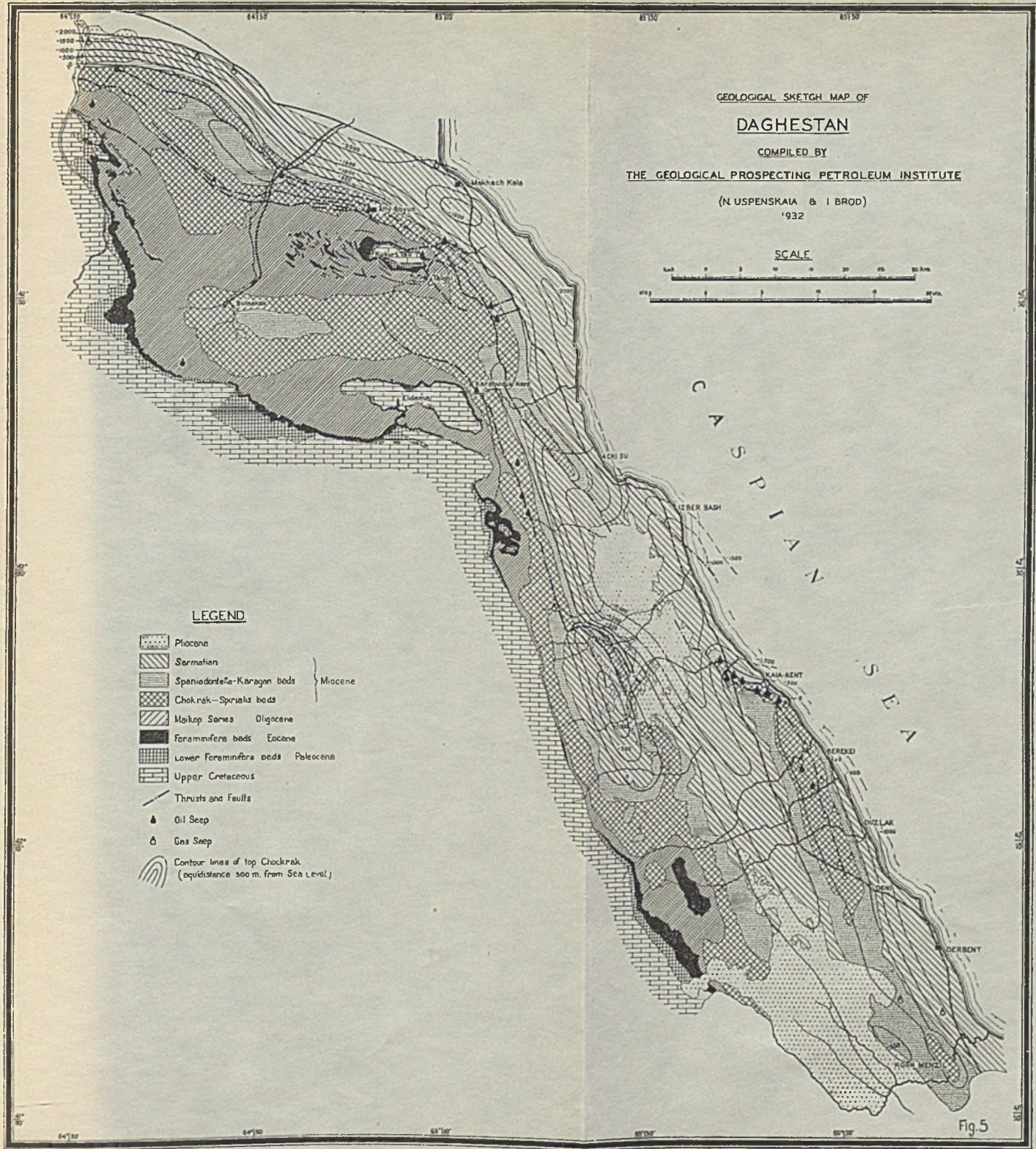


FIG. 5.

the latter may form comparatively gentle structures when compared with the differentially folded overlying beds.

In addition, one has to consider the possible presence of "Blatt-faults." Such a fault, for example, is indicated by a line through Khurdalan-Binagadi-Buzovny accompanied by a row of important mud volcanoes reaching far into Kabristan. Along this line the southern half of Apsheron has probably moved eastwards. At Buzovny an Apsheronian limestone ridge has been displaced to the east for a distance of 1 km.

Evidence of over-riding movements is the displacement of structural highs in formations of different age but belonging to the same area of local uplift. At Kala the anticlinal axis of the Surakhany beds of the Producing Series is situated 3-4 km. to the south-east of the axial region of the younger Apsheronian limestone. The crest of the deeper-seated horizons of the Balakhany beds is still further to the south-east. In the Surakhany field the eastward displacement of deeper horizons is more than 700 m. In addition, the deeper beds show increasingly steeper dips, although the formation belongs to the same cycle of deposition.

On Artem Island the same horizon has been encountered three times during drilling. Even this may be a case of submarine slumping.

Lateral movements of fault blocks coupled with the sliding of entire formations towards the deepest part of the Caspian depression may well account for the existence of the extraordinary ringfolds, bifurcation and even crossing of anticlines as known in Kabristan and Apsheron.

Oilfields.

Fig. 3 shows the distribution of the major folds and oilfields of the Apsheron Peninsula. Of the many structures, the most important is undoubtedly the Kirmaku-Balakhany-Surakhany fold extending to Zyk and Peschany Island. According to Goubkin, this fold may swing from Peschany to Bibi-Eibat. Local uplifts, saddles and normal faults are responsible for the irregular distribution of oil. The northern uplift near the village of Fatmai exposes Eocene beds in its core. The second uplift of Kirmaku is opened to the Spirialis beds. The brachy-anticlines of Balakhany and Sabunchi are closed in the Producing Series. Surakhany, the fourth uplift, is sealed by gas-bearing Apsheronian. Kara Chkhur and Zyk are covered by still younger beds. The Producing Series increases in thickness from north to south—viz. from 1500 to 2500 m. This formation consists of an alternating succession of sands subject to rapid lateral change, intercalated with silts and clays. Although fresh-water fossils are common, the sediments have probably been deposited in estuaries and muddy marine deltas. In the north-eastern Kura plains the Producing Series changes to a fanglomerate, but thins out rapidly towards the main Caucasus Range. Mineralogic examination indicates that the sediments of the Producing Series were deposited both by a Palæo-Volga and an earlier Kura river.

The oilfields of Apsheron are exceptionally rich producers. *Bibi-Eibat* has the biggest acre-yield in the world. Up to eighteen horizons are produced from a thickness of 1850 m. of Producing Series, with an average sand content of 65 per cent. The spacing of wells of 100-120 m. and separate tapping of almost every sand have resulted in the drilling of about

2600 wells. About seventy-four million tons had been produced up to 1st January, 1936. The specific gravity of the oil increases with depth. This is generally the case on the Apsheron fields. The tectonic position of Bibi-Eibat is somewhat outside the normal trend of folds. The presence of mudflows inside the Producing Series probably indicates a deeply seated pseudo-diapiric fold which might be in alignment with the main Kown-Boz-Dag anticline. In this connection the abnormal position of the mud volcano Otman-Boz-Dag demands comment. This volcano probably lies along the prolongation of the Shiki Kaya anticline. In such case the Shiki Kaya anticline would cross the Kara Dag-Utalghi line of uplifts.

Future Prospects.

Undoubtedly many fields remain to be opened up from amongst the 350 structures claimed to have been traced by the Soviet geologists. Drilling for the Cretaceous has already commenced. With Jurassic and Cretaceous source-rocks at depth, any reservoir-rock may yield commercial accumulation of oil. Incongruous folding, "décollement" (Abscherung) of incompetent masses of marls and clays on more rigid limestone and sandstone formations, as well as lateral shifting of axial regions, will probably complicate any prospecting to depth.

Eastern Kura Plain Fields.

These are, in many respects, different from the Apsheronian fields: their alignment is more in accord with the general strike of the western hinterland. The younger sediments are more thickly developed (Apsheronian 800-1000 m., Akchagylian 125 m.). The Producing Series also has increased in thickness, mainly due to additional development of the clays. The discovery of about 300 m. of sand in the Maikop Series of Shiki Kaya (35 km. west of Bibi Eibat), and others in the Spirialis beds, offers hopes for similar conditions in the Kura plain. Besides the Producing Series, oil is also won from Apsheronian beds. As in other oil districts of U.S.S.R., many new fields will be opened up in the Kura flats as exploration proceeds.

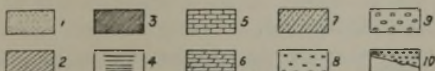
DAGHESTAN REGION.

General Geology.

A cursory review of the general geologic conditions shows the following essential features (see also Fig. 5). The belt of Tertiary sediments along the Caspian Sea has a width of from 30 to 50 km. and a length of about 200 km. The Tertiary includes almost all series from the Palæocene to the Upper Pliocene, attaining a thickness of nearly 4000 m. (see also Fig. 6).

The main orogenic movement took place at the end of the Miocene and beginning of Pliocene. Box-like folds are typical in the region, and are especially common amongst the Mesozoic sediments of mountainous Daghestan. Although the anticlines of the Tertiary belt are relatively wide and flat, they are frequently overthrust to the north, whereby Cretaceous may come to rest on younger Tertiary. Commercial accumulation of oil is mainly associated with the sands of the Miocene Spaniodontella and Chokrak-Spirialis beds. Frequent indications of oil also

Division	Stage	Horizon	Guide-fauna	Thickness in meters	Geologic section
Neogene	Pliocene	Apsheron	Apsheronia graciosa	upto 120	
		Akolaghyll	Cardium dombra	upto 200	
	Sarmatian	Upper	Mastra caspia	100-800 S-N	
		Middle	Cardium fillani	200-750	
		Lower	Cryptomastrea pes anseris	0-400	
			Mastra fragilis	0-100	
			Syndesmia reflexa	150-400 N-S	
		Second Mediterranean stage	Konka	Spinalia Phellax	upto 70
	Karagan		Spanodontella	300-425 S-N	
	Chokrak		Spinalia	400-1100 S-N	
First Mediterranean stage	Tarkhan	Foraminifera	50-70		
	Amussium denudatum	0.5-1.0	Marl		
Paleogene	Oligocene	Upper	Fish Fossils	100-1700 S-N	
		Lower			
	Eocene and Paleocene	Upper Division	Foraminifera	20-60	
		Lower Division	Foraminifera	20-40	
		Lower Division	Foraminifera	20-80	
Upper Cretaceous					



Summarised table of the Tertiary deposits of Daghestan

- 1 - sandstones; 2 - clays; 3 - marly clays; 4 - marly bands; 5 - limestones; 6 - marls
- 7 - marly shale; 8 - siderite concretions; 9 - marly concretions; 10 - basal conglomerate

abundant in the Maikop series and in Eocene marls. Especially rich reservoirs of gas have been encountered in the Maikop series.

Oilfields.

The producing fields will be briefly discussed, as in the accessible literature little is known of some of the comparatively recent developments.

Daghestan Ogni.—This field is located about 13 km. north-west of Derbent. It produces almost pure methane gas from Khadum and Maikop series. Shallow gas accumulation in the quaternary Caspian deposits has been commercially used for a long time for glass manufacture. Similar conditions are found in the dome of *Duzlak*. At *Berekei* oil was produced from the Oligocene Khadum beds and Eocene foraminiferal marls. However, hot iodine- and bromine-containing water flooded the entire field, and even prevented drilling to the Cretaceous. One well produced up to the 50,000 cu. m. per day of this hot water, and is now under control for exploitation of its halogens. The water is derived from Cretaceous, or even Jurassic, beds. The fissure system along which the water travels dissects even incompetent clays, such as those of the Maikop series.

The oil of *Kaia Kent Field* is associated with the Chokrak sands of 30–70 m. in thickness. The production is rarely more than 12 tons per day, but generally less. Nevertheless, it is hoped to sink about 1500 wells to depths of 200–500 m. The Maikop series shows gas and little oil. Whereas Ogni, Duzlak and Berekei belong to one anticline 30 km. in length and about 5 km. in width, the Kaia Kent field is slightly offset to the west and considerably disturbed by thrust faults.

The *Izber Bash Field* is characterized by Chokrak sands of 200 m. thickness, hence wells with an initial production of 200 tons are frequent. A strike fault divides the field. Two-thirds of the prospective area lies in the Caspian Sea. The field was discovered in 1936, and production is derived from Chokrak and Karagan beds. The oil has a specific gravity of 0.834 and carries almost 60 per cent. light fractions. An ultimate production of one hundred million tons is already claimed for this field of scarcely 3½ sq. km. in extent, and this is without taking into consideration any possible prospects of Maikop and Khadum beds or Cretaceous.

The *Achi-Su Field* occurs on a compressed anticline thrust to the south-west—i.e., towards the mountains—similar to Izber Bash. Karagan and Chokrak beds are outcropping in the axial region. The oil is won from the deeper Chokrak sands. The structure is about 20 km. long, and gives promise of developing into a large field.

Future Prospects.

Apart from possible lateral extension of the present fields, there are prospects on other structures for Maikop, Eocene, and especially Cretaceous oil. Oil and gas seeps in the Lower Cretaceous are common.

Slumping and Submarine Sliding.

N. Uspenskaja and I. Brod introduced the excursionists to some most interesting features of submarine sliding that deserve the closest attention. To the west of the village of Karabudakh Kent rises the gently sloping dome of *Eldama*, exposing Upper Cretaceous covered along the flanks by

foraminiferal Palæocene, Eocene, Khadum and Maikop beds (see Fig. 7).

The Cretaceous is represented by well-bedded Senonian marly shales and argillaceous limestone, overlapped by variegated marls and glauconitic sandstone of the lower part of the foraminiferal Palæocene. The brown bituminous argillaceous marls, with remains of the fish *Lyrolepis caucasica*, indicative of younger Eocene age, progressively overstep the Palæocene and Cretaceous, whereby blocks of these whitish, older beds are found completely surrounded by the dark *Lyrolepis* marls. At the same time a small basal conglomerate was formed. Greenish and white marls of the Upper Eocene, 100–200 m. thick, follow, and in their turn over-step the older Eocene and Cretaceous marls. At one place the excursionists noticed large masses of Senonian argillaceous limestone and Palæocene with an "avalanche-like" aspect, rolled together, and completely surrounded by the Upper Eocene foliated marls (see Plate No. 3). The Upper Eocene marls with the twisted and rolled-together slump masses of older beds have glided over strongly crumpled *Lyrolepis* marls. These older beds must have still been in a plastic state when the progressive overlap covered them. "Under-cutting" has probably

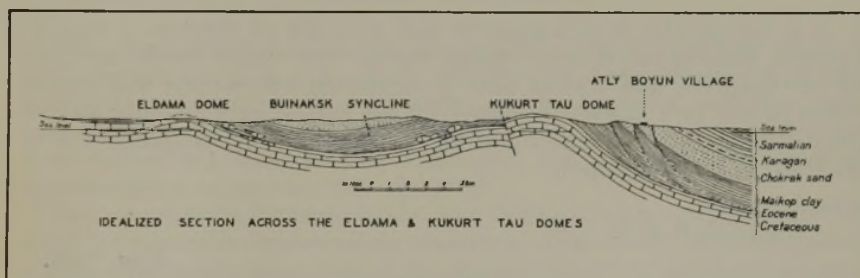


FIG. 7.

taken place below sea level and along a gently dipping slope. Changes of sea current or earthquake accompanying orogenic movements may have assisted in setting such masses in motion.

The submarine sliding reached its maximal dimensions during the deposition of the Miatly zone of the Lower Maikop series. Along the Kukurt Tau Dome, and to the west of Buinaksk, masses of *Lyrolepis* marl, Upper Eocene and Khadum beds, attaining sometimes a dimension of several kilometres in length and several hundred metres in thickness, are found detached from their original position and completely enveloped in the Oligocene areno-argillaceous Miatly beds. These slumped masses were, in earlier days, considered to represent tectonic breccias, overthrust scales or even remains of overthrust sheets. The great change in the thickness of the Maikop series from 1500 m. near the Kukurt Tau dome to 200 m. not far to the south-west of it indicates the presence of somewhat steep submarine slopes during the time of their deposition. The nascent orogenic movement (Pyrenean phase) was probably accompanied by seismic shocks. A combination of these factors has to be considered in determining the cause of this large-scale slumping.

Atly Boyun Pass is interesting on account of the three scales of Chokrak

and Spirialis sandstone thrust towards the south (see also Fig. 7). The shearing took place on the thick series of Maikop shales without having affected the older Eocene and Cretaceous beds. The highly bituminous Maikop series is represented by foliated scaly shales (paper shales) full of fish remains. These shales are considered to be a first-class oil source-rock. Along the joints, sulphur-yellow jarosite powder is common. Thin layers of grey sandstones and siderites are subordinate. Of interest are the so-called "Neptunian dykes" of the Soviet geologists (Plate No. 4). These are sandstone dykes apparently connecting one thin sandstone layer above with a lower one several metres apart. The intervening shales moved after these dykes had been formed and tore them into segments of a "rosary-like" alignment. It is obvious that further movements would completely dislocate the sandstone segments, and the appearance of loose sandstone blocks in the midst of bituminous shale would result, causing a condition which might lead to a variety of false interpretations. The only reasonable explanation offered for the formation of the dykes was that they were squeezed through cracks from one sand-lens to another during compaction.

CONCLUDING REMARKS.

It is not possible in this paper to deal with the oil districts of Grozny, Georgia, Maikop and Kuban. These regions were visited by the members of the Congress, and a great deal of interesting information was gained. Some of the rock samples collected by the author have recently arrived from Russia, and it is hoped that the result of their investigation will be included in a future paper.

*Pointe-a-Pierre,
Trinidad, B.W.I.*

May, 1938.

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THE PURITY OF SOME HIGH-MOLECULAR HYDROCARBONS AS DESCRIBED IN THE LITERATURE.*

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SYNOPSIS.

Attention is directed to the necessity of a more elaborate and systematic research in order to obtain more accurate and fundamental data for high-molecular hydrocarbons of several types.

Stress is laid on the danger of accepting without criticism all data given in the literature. In general, hydrocarbons of which information as to the methods of preparation and a check on the purity are lacking or incomplete should not be accepted as pure without further confirmation.

A consideration of the fundamental and very extensive work of Mikeska has shown that even in this case it is not justifiable to accept all the results without criticism.

It is of importance to know the composition of the natural and artificial hydrocarbon mixtures (*e.g.*, those obtained by polymerization), as the composition determines the properties of the products and also their technical importance. We have in mind particularly the high-molecular hydrocarbon mixtures, such as lubricating oils, of which the chemical examination is as yet but little advanced.

Various methods are used to obtain a better insight into the nature of the high-molecular hydrocarbon mixtures. One group of investigators wishes to ascertain some idea of the composition of the mixtures by preparing a large number of hydrocarbons of different types, and by direct comparison between the properties of the hydrocarbons so prepared and the hydrocarbon mixtures to be examined. However important the knowledge of the properties of a great number of the high-molecular hydrocarbons may be, no final explanation of the structure of the products examined is to be expected for the present when working on these lines. Such a method can be completely satisfactory only after a very large number of pure hydrocarbons has been synthesized; moreover, it must be possible to split the mixtures to be examined completely, or almost completely, into their components. For the present both points entail difficulties; the number of known pure hydrocarbons is small; further, it is particularly unlikely that we shall be able to isolate quantitatively the components from the complex natural and synthetic hydrocarbon mixtures by the means at present at our disposal.

For the present, at any rate, it seems more logical to us to prepare a number of pure hydrocarbons of varying types and to employ these for

* Paper received November 1938. A subsequent paper on this subject will deal with "Preparation of some Pure Hydrocarbons for the Purpose of Testing the Physical Methods in Use for the Examination of Hydrocarbon Mixtures," by H. I. Waterman, J. J. Leendertse and D. W. van Krevelen.

the development and testing of general methods of analysis which may be applied to the hydrocarbon *mixtures* to be examined *as such*, and which give us an idea of the *average composition* of these mixtures. Examples of such analytical methods are the method proposed by Davis and McAllister,¹ the specific refraction-molecular weight method² and the specific parachor-molecular weight method.³ The specific refraction-molecular weight method enables the average elementary composition of the saturated hydrocarbon *mixtures* to be determined quickly and simply (and therefore the average cyclic character which is directly connected with this). The third method gives an indication of the degree of branching of the hydrocarbon mixtures.

Whatever course one wishes to pursue in order to arrive at a clearer insight into the exact composition of higher molecular hydrocarbon mixtures, it will always be important to know the properties of a certain number of pure high-molecular hydrocarbons.

For this reason it is extremely regrettable that so little attention has been paid in the literature to the preparation of these compounds. The data available for high-molecular hydrocarbons are relatively small, and in several cases doubt as to their accuracy is fully justified. Some investigators present their results in such a form that it is quite impossible to decide whether the data are accurate. This incompleteness is often found in the description of the synthesis as well as in the determination of a number of physical constants of the reaction products. Several authors, for example, mention only the melting or boiling point of their substances. An accurate proof of the structure of hydrocarbons described is omitted in many cases, or is incomplete. Existing methods of checking, which had already been developed, are neglected. It is essential that, in view of the difficulties attending the synthesis of pure hydrocarbons, data shall be presented by means of which the purity of the compounds prepared can be critically judged.

As there are always investigators who build on the work of others and reach conclusions from it, without taking into account the possibility of inaccuracies in the synthesis of various compounds carried out by these other investigators, it would in many cases have been better if the data with regard to "pure hydrocarbons" had not been published, as their publication is more likely to hamper the formulation of an exact conception of the composition of high-molecular hydrocarbon mixtures than to contribute towards a solution of the various problems.

Consequently the authors consider that in cases where information as to the method of preparation and a check on the purity are lacking or incomplete, the hydrocarbons may not be accepted as pure without further confirmation.

This lack of accurate data for a number of high-molecular hydrocarbons of different structures gives serious difficulties for the investigation of the structure of natural and synthetic hydrocarbons and mixtures of hydrocarbons. It would be very fortunate if a great number of chemists would give more attention to this important (though very difficult) subject.

Also we must be full of praise for the work of some investigators, who in recent years have felt this lack of data about high-molecular hydrocarbons, and have already attacked this problem in a serious and systematic way.

In this connection, especially, attention may be directed to the very systematic investigations of Mikeska. This author has undertaken the difficult and laborious synthesis of more than fifty high-molecular hydrocarbons. The physical properties of his products have been described in a first paper,⁴ whereas the details of the syntheses will be described in following papers. (A second paper has already been published.⁹) In the meantime in several communications the data of Mikeska have been used.^{5,6,7} The authors were also very glad to find in the work of Mikeska several saturated hydrocarbons, which could be used for checking the specific refraction method mentioned before.²

Unfortunately, however, it was observed that the ultimate composition derived from the specific refraction was not always in accordance with the ultimate composition deduced from the structural formulæ of the hydrocarbons given by Mikeska. Therefore the work of the latter author was studied in more detail. It is especially in connection with the fundamental character of Mikeska's work that the authors feel obliged to publish the conclusions of their investigations in this direction.

In Table I the data of saturated hydrocarbons prepared by Mikeska have been described. The specific refraction, calculated from n_D and d , as well as the "theoretical" specific refraction (calculated from the atomic refraction values of Eisenlohr), have been given. Besides, the number of rings per molecule deduced from the specific refraction is mentioned.

It may be seen that some important discrepancies are observed. Mikeska's compound No. 11 in particular shows very important differences.* As the authors had proved before⁸ that for *mixtures* of hydrocarbons of very divergent types the specific refraction method gives a good idea of the elementary composition of saturated hydrocarbon mixtures within narrow limits, these differences roused some doubt as to the complete accuracy of some of Mikeska's data. Moreover, the deviations observed for Mikeska's compounds are found to show an improbable irregularity. For instance, if we compare compound No. 11 with No. 13, we see in both cases a great difference from the expected value for the specific refraction. However, in a contrary sense, although these two compounds are of exactly the same type, and only differ to an inconsiderable extent in the length of their side-chains.

At most the objection might be raised against the application of the specific refraction method to Mikeska's products that the specific refraction method was specially developed and checked for high-molecular hydrocarbon *mixtures*, whereas Mikeska's article deals with individual substances. We have therefore collected a great number of data from the literature referring to individual high-molecular hydrocarbons. These data are given in Table II.† The differences observed in the elementary com-

* The ultimate composition described by Mikeska is in accordance with the structure accepted by the latter author.

† During the preparation of this paper a very extensive list of physical constants of paraffin hydrocarbons was published by A. V. Grosze and G. Egloff—"The Physical Constants of Paraffin Hydrocarbons," Universal Oil Products Co., Chicago, Ill. Although it is known that not all the compounds mentioned in this paper are pure, so that the data found in the list must be considered with much reservation, Grosze and Egloff's collection can be very useful to the hydrocarbon investigators.

TABLE I.

Formula.	No. ^a	$n_D^{77^\circ F.}$	$d^{77^\circ F.}$	Specific Refraction, 20° C.		Number of Rings per Molecule.		Molecular Weight, Calculated.
				From $n_D^{77^\circ F.}$ and $d^{77^\circ F.}$	Theoretical.	From Specific Refraction.	Theoretical.	
	11	1.4538	0.834	0.3245	0.3294	2.0	1.0	336.38
	12	1.4627	0.8395	0.3278	0.3294	1.4	1.0	392.45
	13	1.4643	0.8327	0.3315	0.3294	0.5	1.0	392.45
	14	1.4677	0.8372	0.3318	0.3294	0.3	1.0	448.51
	37	1.4739	0.863	0.3255	0.3255	2.0	2.0	390.43
	38	1.4759	0.8673	0.3251	0.3260	2.2	2.0	446.50
	39	1.4772	0.8615	0.3280	0.3260	1.4	2.0	446.50
	46	1.4793	0.867	0.3271	0.3262	1.7	2.0	474.53

TABLE II.

Formula.	Molecular Weight.	n_D^a	d_4^b	t^c	Specific Refraction.			Number of Rings.		Literature Cited.
					Experimental.		Theoretical.	From Specific Refraction.	Theoretical.	
					t^c	20° C. (calc.)				
n -Heneicosane	296.36	1.4240	0.7885	70°	0.3364	0.3351	0.3347	0		
n -Tricosane	338.40	1.4288	0.7882	70	0.3355	0.3342	0.3339	-0.1		
n -Methyl-tricosane	338.40	1.4279	0.7862	70	0.3357	0.3344	0.3339	-0.1		
Methyl-didodecyl-methane	366.44	1.4308	0.7720	70	0.3352	0.3339	0.3337	-0.1		
n -Triacontane	394.47	1.4329	0.7759	70	0.3349	0.3336	0.3334	-0.1		
Trimethyl-methane	392.47	1.4329	0.7770	70	0.3342	0.3330	0.3334	-0.1		
n -Triacontane	436.52	1.4348	0.7825	70	0.3346	0.3333	0.3331	-0.1		
n -Hexatriacontane	436.52	1.4367	0.7825	70	0.3358	0.3325	0.3320	-0.1		
n -Heptatriacontane	450.53	1.4367	0.8182	20	0.3352	0.3352	0.3328	-0.7		
n -Doctriacontane	450.53	1.4334	0.7784	72	0.3341	0.3328	0.3328	0	G. Hugel, <i>Chim. et Ind.</i> , 1931, 26,1282.	
n -Heptatriacontane	520.61	1.4328	0.7752	90	0.3351	0.3334	0.3324	-0.3		
n -Heptatriacontane	520.61	1.4348	0.7801	90	0.3344	0.3327	0.3324	-0.1		
n -Octatriacontane	534.63	1.4326	0.7758	90	0.3347	0.3330	0.3323	-0.2	Ref. in J. Müller and S. Pillat, <i>J. Inst. Petr. Techn.</i> , 1935, 21, 887.	
n -Octatriacontane	534.63	1.4376	0.7881	90	0.3329	0.3312	0.3323	0.4		
n - n -Propyl-pentatriacontane	534.63	1.4376	0.7881	90	0.3329	0.3312	0.3323	0.4		
Perhydroqualene	422.49	1.4535	0.8093	20	0.3342	0.3342	0.3331	-0.3	I. M. Heilbron, T. P. Hilditch and E. D. Mann, <i>J. Chem. Soc.</i> , 1926, 3131.	
16-Methyl-hentriacontane	450.53	1.4517	0.8096	40.5	0.3330	0.3325	0.3329	0.1	H. Sulda and R. Planckh, <i>Ber.</i> , 1933, 66, 1445.	
16-Ethyl-hentriacontane	464.55	1.4583	0.8211	23	0.3325	0.3321	0.3328	-0.1		
16-Butyl-hentriacontane	492.58	1.4579	0.8206	25	0.3324	0.3323	0.3327	-0.1		
2 : 6 : 10 : 14 : 19 : 23 : 27 : 31-Octamethyl-dodecatricontane	502.66	1.4584	0.8222	18	0.3322	0.3322	0.3321	0	P. Karrer, A. Helfenstein, B. Pieper and A. Wetstein, <i>Helv. Chim. Acta</i> , 1931, 14, 495.	
4 : 8 : 12 : 16 : 20 : 24 : 28 : 32 : 36-Octamethyl-octatriacontane	646.76	1.46265	0.8287	20.9	0.3321	0.3321	0.3318	-0.1	P. Karrer, M. Stoll, Ph. Stevens, <i>ibid.</i> , 1931, 14, 1194.	

TABLE II—continued.

Formula.	Molecular Weight.	n_D^t .	d_4^t .	$t^\circ C.$	Specific Refraction.			Number of Rings.		Literature Cited.
					Experimental.		Theoretical.	From Specific Refraction.	Theoretical.	
					$t^\circ C.$	$20^\circ C.$ (calc.).				
$CH_3CH(CH_2)_2CH(CH_2)_2CH(CH_2)_2CHCH_3$	$C_{16}H_{32}$	1.4382	0.7853	25°	0.3344	0.3343	0.3348	0.1	0	F. G. Fischer and K. Löwenberg, <i>Ann.</i> , 1929, 475, 204.
$CH_3(CH_2)_2CH(CH_2)_2CH(CH_2)_2CH(CH_2)_2CH_3$	$C_{18}H_{36}$	1.44937	0.8027	19.8	0.3344	0.3344	0.3352	0.1	0	P. Karrer, Th. Goldt, <i>Helv. Chim. Acta</i> , 1930, 13, 707.
$CH_3CH(CH_2)_2CH(CH_2)_2CH(CH_2)_2CH_3$	$C_{14}H_{28}$	1.4502	0.8054	20	0.3338	0.3338	0.3339	0	0	R. Kuhn and L. Ehmann, <i>ibid.</i> , 1929, 12, 904.
Tetramethylisocane $(CH_3)_2CH(CH_2)_2CH(CH_2)_2$	$C_{14}H_{28}$	1.43023	0.7728 (calc.)	13.85	0.3344	0.3346	0.3372	0.3	0	S. Landa and A. Keivan, <i>Trav. Chim. Technologique</i> , 1931, 3, 367.
$CH_3(CH_2)_2CH(CH_2)_2$	$C_{11}H_{22}$	1.43942	0.7845 (calc.)	13.9	0.3356	0.3358	0.3353	-0.1	0	S. Landa and R. Riedl, <i>ibid.</i> , 1930, 2, 520.
$CH_3CH_2CH(CH_2)_2CHCH_3$	$C_{11}H_{22}$	1.44473	0.7956 (calc.)	15.2	0.3343	0.3344	0.3353	0.2	0	S. Landa and M. Habada, <i>ibid.</i> , 1936, 8, 473.
$C_2H_5CH(CH_2)_2CH_3$	$C_{10}H_{20}$	1.4476	0.8014 (calc.)	10.6	0.3338	0.3340	0.3347	0.1	0	S. Landa and V. Silva, <i>ibid.</i> , 1932, 4, 538.
$C_2H_5CH(CH_2)_2CH_2$	$C_{10}H_{20}$	1.44855	0.8011 (calc.)	14.0	0.3345	0.3346	0.3342	-0.1	0	S. Landa, J. Čech and V. Silva, <i>ibid.</i> , 1933, 5, 204.
$C_4H_9CH(CH_2)_2CH_3$	$C_{14}H_{28}$	—	—	—	0.3337	—	0.3339	0	0	
$C_4H_9CH_2CH(CH_2)_2CH_2$	$C_{14}H_{28}$	1.46620	0.8808 (calc.)	17.5	0.3261	0.3262	0.3256	1.8	2	
$C_4H_9CH_2CH_2CH(CH_2)_2CH_2$	$C_{16}H_{32}$	1.49111	0.8892 (calc.)	15	0.3258	0.3259	0.3257	1.9	2	S. Landa and J. Čech, <i>ibid.</i> , 1934, 6, 423.
$C_4H_9CH_2CH_2CH_2CH_2CH_2$	$C_{16}H_{32}$	1.4612	0.8520	20	0.3299	0.3299	0.3204	0.9	1	A. J. Haasen Smit, <i>Proc. Kon. Academie van Wetenschappen, Amsterdam</i> , 1931, 34, 165.
$C_4H_9(CH_2)_2CH_2$	$C_{10}H_{20}$	1.48583	0.8860	17.3	0.3239	0.3240	0.3257	2.5	2	P. Karrer, H. Salomon, R. Meier and O. Walker, <i>Helv. Chim. Acta</i> , 1932, 15, 878.

3: 8-Dimethyl-1: 10-di-(1': 1': 3'-tri-methyl-2'-cyclohexyl)-decane

TABLE II—continued.

Formula.	Molecular Weight.	n_D^t .	d_4^t .	$t^\circ \text{C.}$	Specific Refraction.			Number of Rings.		Literature Cited.
					Experimental.		Theoretical.	From Specific Refraction.	Theoretical.	
					$t^\circ \text{C.}$	20°C. (calc.).				
$\text{C}_4\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	140.16	1.4556	0.8305	16°	0.3271	0.3272	0.3294	1.2	1	O. M. Halse, <i>J. für Pract. Chemie</i> , 1915, 92, 40.
$\text{C}_4\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	154.18	1.4538	0.8226	16	0.3291	0.3292	0.3294	1.0	1	
$\text{C}_4\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	168.19	1.4670	0.8372	16	0.3314	0.3315	0.3294	0.7	1	
$\text{C}_4\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	168.19	1.4574	0.8310	16	0.3280	0.3281	0.3294	1.1	1	
$\text{C}_6\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	182.21	1.4658	0.8388	16	0.3301	0.3302	0.3294	0.9	1	
$\text{C}_6\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	182.21	1.4580	0.8304	16	0.3286	0.3287	0.3294	1.1	1	
$\text{C}_6\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	182.21	1.4646	0.8406	16	0.3286	0.3287	0.3294	1.1	1	
$\text{C}_6\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	196.22	1.4685	0.8484 (calc.)	23	0.3280	0.3279	0.3294	1.2	1	
$\text{C}_6\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	196.22	1.4717	0.8483	19	0.3299	0.3299	0.3294	0.9	1	
$\text{C}_6\text{H}_{11}-\text{C} \begin{array}{l} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{CH}_2 \\ \\ \text{CH}_3 \end{array}$	196.22	1.4754	0.8519 (calc.)	23	0.3307	0.3306	0.3294	0.8	1	

TABLE II—continued.

Formula.	Molecular Weight.	n_D^t .	d_4^t .	$t^\circ \text{C.}$	Specific Refraction.			Number of Rings.		Literature Cited.
					Experimental.		Theoretical.	From Specific Refraction.	Theoretical.	
					$t^\circ \text{C.}$	20°C. (calc.).				
C_8H_{11} — $\begin{array}{c} \text{CH}_2 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_{11} \end{array}$	210.24	1.4871	0.8689 (calc.)	23°	0.3311	0.3310	0.3294	0.8	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_{11} \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_{11} \end{array}$	210.24	1.4598	0.8348 (calc.)	23	0.3280	0.3279	0.3294	1.2	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_9 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_9 \end{array}$	224.26	1.4622	0.8368 (calc.)	23	0.3287	0.3286	0.3294	1.1	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_9 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_9 \end{array}$	224.26	1.4606	0.8354 (calc.)	23	0.3282	0.3281	0.3294	1.2	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_7 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_7 \end{array}$	238.27	1.4658	0.8413 (calc.)	23	0.3291	0.3290	0.3294	1.1	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_7 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_7 \end{array}$	252.29	1.4666	0.8444 (calc.)	20	0.3284	0.3284	0.3294	1.2	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_5 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_5 \end{array}$	266.31	1.4789	0.8685 (calc.)	20	0.3265	0.3265	0.3294	1.5	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_3 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_3 \end{array}$	280.32	1.4646	0.8425 (calc.)	20	0.3279	0.3279	0.3294	1.3	1	
C_8H_{11} — $\begin{array}{c} \text{C}_7\text{H}_1 \\ \\ \text{C} \\ \\ \text{C}_7\text{H}_1 \end{array}$	294.34	1.4905	0.8801 (calc.)	20	0.3288	0.3288	0.3294	1.1	1	

O. M. Halse, *J. für Pract. Chemie*, 1915, 92, 40.

TABLE II.—continued.


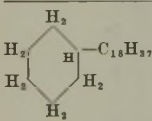
Formula.	Molecular Weight.	n_D^t .	d_4^t .	$t^{\circ} C.$	Specific Refraction.			Number of Rings.		Literature Cited.
					Experimental.		Theoretical.	From Specific Refraction.	Theoretical.	
					$t^{\circ} C.$	$20^{\circ} C.$ (calc.).				
<i>cyclo</i> Hexane	C ₆ H ₁₂	1.4268	0.7783	20°	0.3297	0.3297	0.3294	1.0	1	F. Eisenlohr, <i>Fortschritte der Chem.</i> , 1924—26, 18, 540.
<i>cyclo</i> Octane	C ₈ H ₁₆	1.4572	0.8332	16	0.3270	0.3271	0.3294	1.2	1	{ L. Ruzicka, M. Stoll, H. W. Huyser and H. A. Boekennoogen, <i>Helv. Chem. Acta</i> , 1930, 13, 1152.
<i>cyclo</i> Tetradecane	C ₁₄ H ₂₈	1.4532	0.8284	75	0.3264	0.3250	0.3294	1.6	1	
<i>cyclo</i> Pentadecane	C ₁₅ H ₃₀	1.4553	0.828	71	0.3279	0.3266	0.3294	1.4	1	{ L. Ruzicka and H. A. Boekennoogen, <i>ibid.</i> , 1931, 14, 1319.
Methylcyclopentadecane	C ₁₆ H ₃₂	1.4743	0.8594	18	0.3272	0.3272	0.3294	1.3	1	
Dicyclooctyl	C ₁₄ H ₃₀	1.5018	0.9277	20	0.3180	0.3180	0.3225	2.7	2	
<i>cyclo</i> Hexadecane	C ₁₆ H ₃₂	1.4557	0.824	72	0.3297	0.3284	0.3294	1.1	1	{ L. Ruzicka, M. Stoll, H. W. Huyser and H. A. Boekennoogen, <i>ibid.</i> , 1930, 13, 1152.
<i>cyclo</i> Heptadecane	C ₁₇ H ₃₄	1.4435	0.8062	101	0.3292	0.3272	0.3294	1.3	1	
<i>cyclo</i> Octadecane	C ₁₈ H ₃₆	1.4532	0.824	72	0.3288	0.3275	0.3294	1.3	1	{ L. Ruzicka and H. A. Boekennoogen, <i>ibid.</i> , 1931, 14, 1319.
<i>cyclo</i> Docosane	C ₂₂ H ₄₄	1.4498	0.8174	75	0.3286	0.3272	0.3294	1.4	1	
<i>cyclo</i> Hexacosane	C ₂₆ H ₅₂	1.4490	0.8120	78	0.3303	0.3288	0.3294	1.1	1	{ L. Ruzicka and H. A. Boekennoogen, <i>ibid.</i> , 1931, 14, 1319.
<i>cyclo</i> Triacontane	C ₃₀ H ₆₀	1.4547	0.8213	73	0.3302	0.3289	0.3294	1.1	1	
Dicyclopentadecyl	C ₃₀ H ₅₈	1.5002	0.9104	20	0.3232	0.3232	0.3250	2.6	2	
<i>cyclo</i> Tricosane	C ₂₇ H ₅₄	1.4558	0.8300	56	0.3274	0.3265	0.3294	1.6	1	L. Ruzicka and M. Stoll, <i>ibid.</i> , 1933, 16, 493.
1:16-Dimethylcyclotridecane	C ₂₃ H ₄₆	1.4498	0.813	80	0.3304	0.3289	0.3294	1.1	1	L. Ruzicka and H. A. Boekennoogen, <i>ibid.</i> , 1931, 14, 1319.
<i>cyclo</i> Dodecacontane	C ₃₃ H ₆₆	1.4568	0.8261	70	0.3295	0.3283	0.3294	1.3	1	{ L. Ruzicka, M. Hürbin and M. Furter, <i>ibid.</i> , 1934, 17, 78.
<i>cyclo</i> Tetracontane	C ₃₄ H ₆₈	1.4554	0.8229	76	0.3300	0.3280	0.3294	1.3	1	

From the refractometric observations of Ruzicka c.s. for the helium lines τ and f are calculated the n_D values with the formula: $n_D = n_f - 0.026(n_f - n_p)$ (N. Schoorl, "Organische Analyse," 1937, third impression, page 45).

position calculated from the observations and those found are again expressed here in the cyclic character of the products. Although the cyclic character expected does not always tally completely with that found, the deviations are small for the majority of the compounds : a deviation of one ring per molecule, as found in one case with Mikeska's compounds was nowhere encountered. Admittedly this does not constitute absolute proof that the specific refraction method can also be applied off-hand to individual high-molecular compounds, the more so as the purity of these compounds certainly cannot invariably be guaranteed. Moreover, the refractive index and the density were in several cases not determined at the same temperature, so that a conversion was required in our calculations. Still, the general impression of the figures in Table II supports our opinion that the specific refraction method can also be employed within rather narrow limits for judging high-molecular hydrocarbons as such.

Finally we decided, on the basis of these considerations, to put our views to the test by preparing in a pure condition, and in a manner different from that adopted by Mikeska, two of the compounds which had been prepared by Mikeska, and the properties of which led us to assume an inaccuracy in the preparation or in the data given. The complete description of this preparation and of the properties of the products will be given in a second and third paper.* Table III shows only those details which are of importance for this article.

TABLE III.

Formula.	Specific Refraction.			Number of Rings per Molecule.		Remarks.
	t° C.	Observed.	Theoretical.	From Specific Refraction.	Theoretical.	
 $C_{18}H_{17}$	20°	0.3313 (extrap.)	—	—	—	Our preparation.
	25°	0.3333	—	—	—	Preparation of Mikeska.
 $C_{18}H_{17}$	20°	0.3297 (extrap.)	0.3294	0.9	1.0	Our preparation.
	25°	0.3246	0.3294	2.0	1.0	Preparation of Mikeska. ⁴

On considering the results, it was found, indeed, that the data given by Mikeska for his product 11 do not tally with the data observed for our preparation $C_6H_{11-n}(C_{18}H_{37})$ (prepared by us in two independent ways). Our product possesses the properties which the structure would lead us to expect. The difference in the number of rings found from the specific refraction and from the structural formula is wholly within the limit of the universally accepted error of the specific refraction method.

Therefore we must conclude that Mikeska's compound 11 cannot indeed

* The investigations were carried out by D. W. van Krevelen and J. F. Sirks.

have been pure $C_6H_{11-n}(C_{18}H_{37})$. It should be noted that Mikeska's ultimate analyses do not agree with our results. However, in view of our considerations already described, especially in connection with our own synthesis of the hydrocarbon in two independent ways, it is very difficult for us to question the accuracy of our own specific refraction data for the compound $C_6H_{11-n}(C_{18}H_{37})$. In any case, it may be seen from the results previously described that even the serious investigations of Mikeska cannot be accepted in all respects without a further detailed research. This stresses once more the necessity of systematic investigations by a great number of chemists in order to obtain accurate data for high-molecular hydrocarbons of several types.

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Delft.*

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NOTE ON THE DETERMINATION OF NEUTRAL OIL IN "SODIUM SULPHONATES."*

By E. J. BOORMAN, B.Sc., Ph.D., A.R.C.S., D.I.C.

SYNOPSIS.

The neutral oil present in "sodium sulphonates" is obtained substantially free from contamination by extraction with petroleum spirit from a solution of the sample, freed from inorganic salts, in a mixture of equal volumes of water, glycerol and alcohol; and washing the extracts with 85 per cent. by volume aqueous alcohol.

THE "sodium sulphonates" of commerce, obtained during the refining of petroleum oils with oleum, usually consist of the alkali salts of sulphonic acids, with minor proportions of naphthenic acids or their salts, inorganic salts, free alkali, water and hydrocarbon oil. The alkali sulphonates are readily soluble in diethyl ether or in petroleum spirit, and may be extracted from their aqueous solutions by these solvents, particularly in the presence of free alkali or water-soluble inorganic salts. Hence, the usual methods of separation of neutral from acidic material by extracting the former from alkaline aqueous solution fail, owing to partial or complete intermiscibility of the two layers.

The method described herein overcomes this difficulty, and while v. Pilat and Sereda,¹ W. Schaeffer,² and others have been concerned with a complete analysis of these materials, the present method is confined to the determination of the neutral oil in the presence of large amounts of sulphonates. It is conducted in two stages:

(i) The sample is first "parted" between petroleum spirit and a strongly acid solution in aqueous alcohol (the presence of the latter facilitates separation). The inorganic matter (a cause of erratic solubility) passes into the dilute alcoholic layer, and the free sulphonic acids, with the neutral oil, pass into the petroleum spirit layer.

(ii) The acidic material from the petroleum spirit extract is neutralized with sodium hydroxide and dissolved in a mixture of equal volumes of water, alcohol (industrial methylated spirit) and glycerol. This mixture contains also the neutral oil, and is extracted with petroleum ether. This extract is practically free from sodium salts, final purification being effected by washing it with aqueous alcohol. The extract is evaporated to constant weight and the residual oil weighed.

If desired, the petroleum spirit extract containing both acids and oil may be evaporated to constant weight. The proportion of acidic material may be estimated by difference after the determination of the oil.

No samples of the pure acids were available, and so no direct check on

* Paper received December 8th, 1938.

the accuracy of the method was possible. Varying quantities of the same sample were submitted to the procedure, and found to give consistent results, indicating that there is no interference by partial solubility of sodium salts, as this would be expected to vary with the amount of sample used. This had, in fact, been found to be the case during trials of less successful methods. Further checks were obtained in many cases by blending the sample with purified heavy mineral oil ("white oil") and analysing the mixture. The results obtained agreed with those calculated for the blend. Finally, the oil obtained was ignited and the ash determined. In the case of the oil from a single analysis, this was negligible, but ashing of the accumulated oil from several series of experiments yielded a very small percentage of ash, which appeared to be sodium sulphate, indicating that a small amount of sodium sulphonates were passing into the final extract. The quantity, however, appears to be negligible.

METHOD.

Approximately 5 gm. of the sample are weighed, correct to 0.001 gm., into a small beaker, and dispersed by stirring in a mixture of 10 ml. of concentrated hydrochloric acid and 20 ml. of industrial methylated spirit (I.M.S., sp. gr. 0.8210). The suspension is washed into a Stokes tube of the type described in report No. 4 on milk products,³ with petroleum spirit, and extracted three times with quantities of 30–35 ml. of this solvent. There is a tendency for emulsions to form, but slight centrifuging readily causes clean separation. The combined extracts are evaporated, on the steam-bath if necessary, to constant weight (1–1½ hr.). Prolonged heating causes darkening, and should be avoided. Three extractions are found to suffice.

To the residue so obtained are added 20 ml. of a mixture of equal volumes of glycerol and I.M.S., and sufficient solid phenolphthalein to yield a visible colour when made just alkaline by titration with *N* sodium hydroxide solution (say x ml.); $10-x$ ml. of water are added, and the whole is washed into a Stokes tube with petroleum ether, and extracted three times with 30–35 ml. quantities of the latter. In some cases a very thick emulsion forms, which cannot be broken by centrifuging; but if this is allowed to stand for 10–15 min., clean separation is obtained. Each extract is washed with the same 30-ml. quantity of aqueous alcohol (85 per cent. by volume I.M.S.) in a second Stokes tube; the combined extracts are evaporated to constant weight in a tared flask, and the oil is weighed (any emulsions which may form during this washing are broken by centrifuging, but this is rarely necessary). This is a clear, amber or pale-brown liquid. There seems to be no loss of oil by retention in either of the aqueous layers, as continued extraction produces no further oil.

A typical series of results on a sample of residue, both alone and blended with "white oil," is given on p. 102.

A total of forty-eight analyses on various samples yielded results differing by not more than ± 0.2 per cent. from the mean for the relevant sample.

The oil from several analyses was collected and the ash determined. This was 0.07 per cent. on the oil, or 0.025 per cent. on the sample, and

Wt., gm.	Oil found, %.	Mean, %.	Blend with Oil.		
			Wt., g.	Oil, %.	
				Calc.	Found.
5.016	36.7	36.8	5.115	50.0	50.2
4.054	36.8		5.063	62.2	62.3
2.935	36.9		5.018	74.5	74.4

appeared to be sodium sulphate. In subsequent experiments, as a qualitative check, the oil from a set of analyses was ashed, the residue dissolved in dilute hydrochloric acid, and tested with barium chloride solution. In some cases no precipitate was formed, and at the most an opalescence after standing was obtained, indicating that the impurities (sodium salts) were negligible. Further, the oil so obtained was found to be neutral (to phenolphthalein), indicating that the acids are quantitatively retained in the alkaline solution. The oil contained less than 0.1 per cent. (0.06 per cent.) sulphur, showing it to be essentially free from sulphur compounds.

The author is indebted to Mr. F. S. Aumonier for valuable criticism, and to Dr. J. J. Fox, C.B., Government Chemist, for permission to publish this work.

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- ³ *Analyst*, 1936, **61**, 105.

FIRE PRECAUTIONS IN A PETROLEUM LABORATORY.*

By J. S. JACKSON, B.Sc., F.I.C. (Member).

FIRE precautions are of obvious importance in any chemical laboratory, but they become of vital importance in any laboratory handling petroleum products. The experience gained in the Shell Laboratories, London, during the past few years in this connection should therefore be of interest. While no serious fire has occurred, a number of small accidental fires have taken place, any one of which could of course have resulted in a major conflagration. The matter was therefore studied closely, and a scheme was evolved by which the person responsible for the fire was asked to report immediately the full circumstances in which the fire occurred. Special efforts were made to remove any element of fear and any suggestion or suspicion of disciplinary action. In other words, every effort was made to secure the operators' full confidence and co-operation, and no difficulty has been experienced in securing the following information from the person concerned :

Location of fire.
Date , time of day.
Product involved.
Person involved.
Description of fire.
Duration of fire.
How extinguished.
Cause of fire.
Extent of damage (if any) and whether the fire
was due to negligence.

The scheme has worked exceedingly well, and it has been possible to study the actual circumstances causing small fires and, wherever possible, such causes have been eliminated. A few actual examples will indicate the working of the scheme and the results attained.

In the I.P.T. method for estimating water in petroleum products, the use of a gas-burner for heating the flask is not excluded. Where an ordinary bunsen flame was used, it was found that puking frequently occurred, causing inflammable solvent to escape from the top of the condenser. This was, of course, ignited by the bunsen, and a small but dangerous fire ensued. Electric heaters which are practically flame-proof have been designed for use in this test.

For a long time it was common practice to clean viscometers, such as the Redwood Viscometer, with inflammable solvents. This led to

* Paper received November 9th, 1938.

occasional minor fires, and finally the use of carbon tetrachloride was standardized for this purpose.

In the apparatus specified by the B.S.I. for the determination of the volatility of transformer oils, it is possible for the condenser to be so placed that it may become choked with liquid, and subsequently overflow. Three minor fires were traced to this defect, and it was a simple matter to arrange that the condenser could not be inserted beyond a certain predetermined safe point. Here again it was thought desirable to design an electric heater shaped to fit the curved bottom of the apparatus.

From time to time fires have been caused by bunsens being allowed to "burn back" as the result of the bunsen tube having become kinked. Accordingly, it has been made standard practice not to use any tube other than the special uninkable tube marketed by one of the leading gas companies.

For work involving the use of inflammable solvents special benches have been constructed and set apart. These are known as "no lights" benches, and the risk of inflammable vapours being ignited has been greatly reduced. These benches are not equipped with either gas or electricity.

A great deal of difficulty has been experienced in handling large separating funnels containing inflammable solvents, and an occasional accident has led to the danger of a serious fire. Here again special benches have been set aside for such work, and special racks have been constructed for holding these separating funnels when they are not actually being handled. Otherwise such separating funnels are normally held in various forms of clamp in a very precarious fashion.

Lately special green-ribbed chest bottles have been used as containers for all inflammable solvents. The capacity of these bottles may not exceed 500 mls., and they differ in colour and shape from any other bottles in use in the Laboratories. It is therefore possible to see at a glance whether any inflammable solvents are within reach of naked flames, and for operators to know immediately when they are handling an inflammable solvent. When not in use, all such inflammable solvents are stored in metal cabinets, the shelves of which are divided into compartments, each taking only one bottle. In addition to distinctive labels, a scheme of coloured designs—squares, diamonds, circles, etc.—is used to indicate the different solvents. The design on the bottle is repeated on the stopper to avoid as far as possible the loss or interchange of stoppers. This scheme has recently been elaborated by the introduction of special coloured wash-bottles, which may only be used for inflammable solvents.

In general, every effort has been made to eliminate the use of naked flames or exposed electric elements in all work involving the use of inflammable substances, and in converting apparatus to electric heating care has been taken to use enclosed elements, and to render the whole apparatus as nearly flame-proof as possible.

In each laboratory a satisfactory supply of fire-fighting materials is maintained. This includes such materials as sand, foamite and fire blankets. Recently the use of cylinders of carbon dioxide which deliver finely divided CO_2 snow has been found exceedingly effective. In each section of the laboratory at least two men have been appointed as fire-

men, and in case of any fire the essential instructions are that all other members of the staff should withdraw, taking with them any inflammable or dangerous material that can be removed easily and safely. The fire is then dealt with by a limited number of trained, skilled men. This arrangement is designed to eliminate misdirected effort and any danger of panic. Experience has frequently shown that the most urgent necessity is to prevent the fire being spread by misdirected effort on the part of excitable people.

SECONDARY REFERENCE FUELS FOR USE IN THE DETERMINATION OF OCTANE NUMBERS.

REPORT by Chemical Standardization Committee (Knock-Rating of Aviation Fuels Panel) of the Institute of Petroleum.

ALTHOUGH *n*-heptane and *iso*-octane are the fundamental standards of the octane scale of knock-rating, it is preferable, in order to secure the best possible degree of reproducibility and correlation between different laboratories and different C.F.R. engines, to use secondary reference fuels which have been authoritatively calibrated against the primary standard hydrocarbons in a large number of C.F.R. engines.

Three such standardized secondary reference fuels, known as the A, C and F series, are obtainable,* and their calibration curves in terms of octane number (Motor Method) are issued with the authority of the C.F.R. Committee.

For rating leaded fuels above the octane number for C the Institute of Petroleum Standardization Committee (Knock-Rating Panel) recommends the use of reference fuel C and lead tetra-ethyl up to 4 mls. per Imperial gallon in preference to C.11 and F.1 blends. This covers the range of about 79 to 93 octane number. A calibration curve for blends of C.11 and lead tetra-ethyl against the primary reference fuels has been prepared by the C.F.R. Exchange Group, and is issued by the Standard Oil Development Company on request when ordering C.11 fuel. (This curve has not been issued officially by the C.F.R. Committee.)

The Institute of Petroleum, through its Knock-Rating of Aviation Fuels Panel, has recently conducted further calibration tests on these secondary reference fuels on which to base recommendations concerning the knock-rating of aviation fuels.

Calibration curves have also been prepared for blends of C and F secondary reference fuels containing a constant concentration of lead tetra-ethyl; these curves are suitable for the rating of motor and aviation fuels containing lead tetra-ethyl of over 92 octane number. The first stage of this work was carried out with 19 C.F.R. engines and the second stage with 17 engines.

The Standardization Committee of the Institute of Petroleum authorizes the publication of the following recommendations resulting from this investigation :—

A. Calibration Curves for Secondary Reference Fuels A.5–C.10 and A.5–C.11 by the Aviation Method (I.P.T. G.39a (T)).

The average calibration curves for these two pairs of fuels determined by the Aviation Method (I.P.T. G.39a (T)) differ by less than 0.1 octane number from the Motor Method calibration curves issued by the C.F.R. Committee for these fuels. These latter curves may therefore be used

* These fuels are manufactured by the Standard Oil Company of New Jersey, and may be ordered from the Standard Oil Development Company, Elizabeth, New Jersey.

without modification for knock-rating according to Aviation Method I.P.T. G.39a (T).

B. Calibration Curves for Leaded Secondary Reference Fuels F.1-C.10 and F.1-C.11 by the C.F.R. Motor Method (I.P.T. G.39 (T)).

The following calibration curves (Figs. 1 and 2) * are approved as suitable for use in determining the knock-rating by Motor Method of those

CALIBRATION CURVE. "C.F.R. MOTOR METHOD." (I.P.T. G.39 (T).)

Approved by The Institute of Petroleum, London, December, 1938.

Blends of Standard Reference Fuels.

F.1 + 4 mls. Tetra-ethyl Lead per Imperial Gln.

and

C.10 + 4 mls. Tetra-ethyl Lead per Imperial Gln.

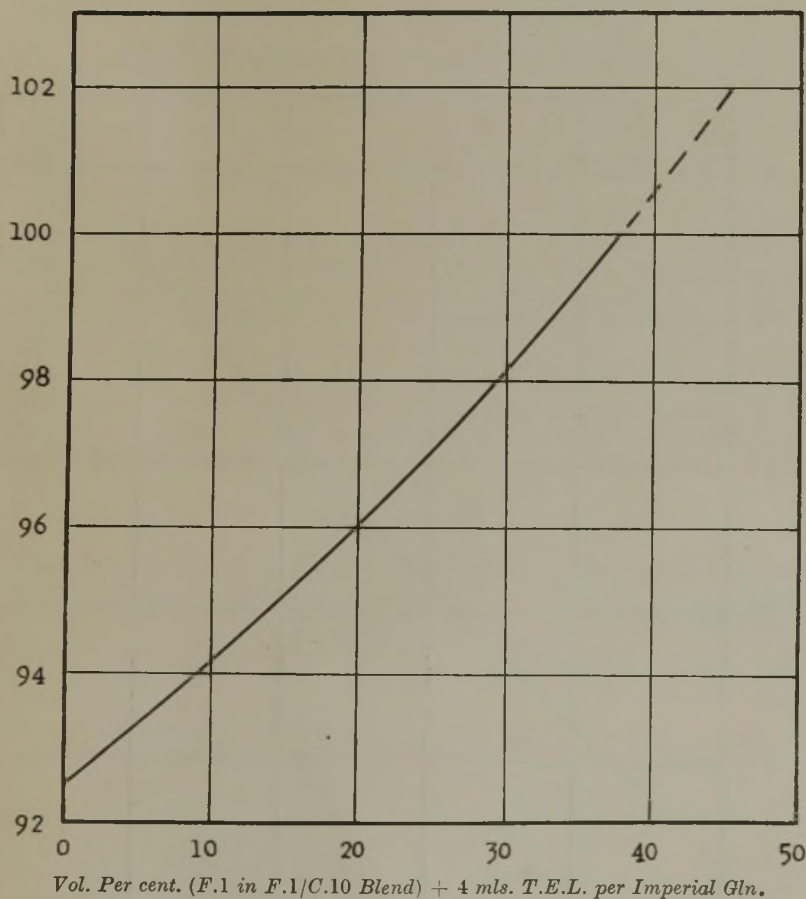


FIG. 1.

* Copies of these curves for laboratory use may be obtained on application to The Secretary, The Institute of Petroleum, The Adelphi, London, W.C.2.

motor or aviation fuels containing lead tetra-ethyl which fall within the range of the curves.

Fig. 1 is based on fuel blends prepared from Secondary Reference Fuel F.1 plus 4 mls. lead tetra-ethyl per Imperial gallon and Secondary Reference Fuel C.10 plus 4 mls. lead tetra-ethyl per Imperial gallon and covers octane numbers of 93 and upwards.

Fig. 2 is based on fuel blends prepared from Secondary Reference Fuel F.1 plus 4 mls. lead tetra-ethyl per Imperial gallon and Secondary Reference Fuel C.11 plus 4 mls. lead tetra-ethyl per Imperial gallon and covers octane numbers of 93 and upwards.

CALIBRATION CURVE. "C.F.R. MOTOR METHOD." (I.P.T. G.39 (T).)

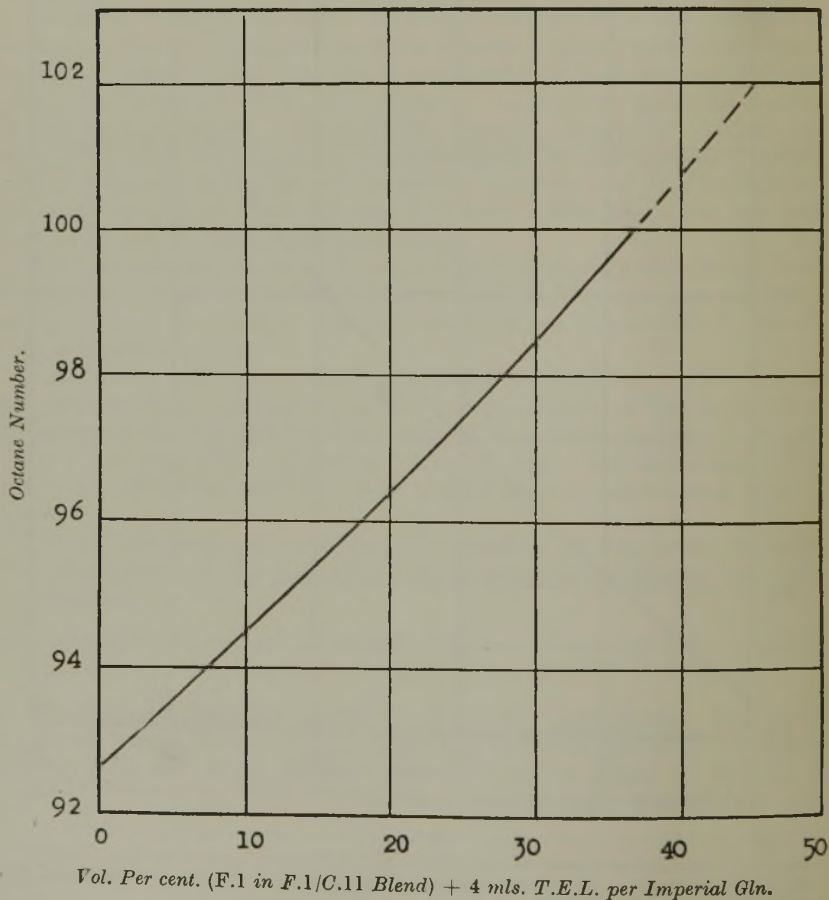
Approved by The Institute of Petroleum, London, December, 1938.

Blends of Standard Reference Fuels.

F.1 + 4 mls. Tetra-ethyl Lead per Imperial Gln.

and

C.11 + 4 mls. Tetra-ethyl Lead per Imperial Gln.



Vol. Per cent. (F.1 in F.1/C.11 Blend) + 4 mls. T.E.L. per Imperial Gln.

FIG. 2.

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Geology.

87. Michigan's Allegan Boom. N. X. Lyons. *Oil Wkly*, 7.11.38, **91** (9), 42.—Allegan and Van Buren counties lie on the south-western edge of the Michigan Basin. The structures in this area do not always conform to the general north-west to south-east trends in the main basin, and several theories have been put forward to explain this anomaly, but so far none of them has been proved.

Three structural trends have been discovered, and it is suspected that cross-faulting is present. Production is from the Traverse Limestone, and the Salem, Monterey and Dorr fields all produce from this horizon. Accumulation has taken place in the upper part of the limestone just under the contact of the overlying Antrim Shale. The porosity of the reservoir rock varies greatly, and this factor influences production to a large extent. J. A. G.

88. Lease Play Gains Momentum as Deep Wildcats in Missouri Start. D. Dalrymple. *Oil Gas J.*, 10.11.38, **37** (26), 18-19.—The area of North-East Missouri is one that may contain undiscovered oilfields. The regional geology is represented by the Nemo Uplift on the east and a similar but higher uplift on the west. Resting on the basement between these two features are a series of limestones, sandstones and shales which form an anticlinal arch trending east-west and dipping in a north-westerly direction. The Lincoln Fold which trends north-west from the Missouri River is considered to be of great importance. Around its southern end erosion has removed the younger rocks, but farther north the Mississippian is present, and below this series the St. Peters sand lies at depths ranging from 200 to 1300 ft. The total thickness of sediments varies from 2500 to 3000 ft. J. A. G.

89. Geology of Allegan County and the Surrounding District in South-Western Michigan. R. B. Newcombe. *Oil Gas J.*, 1.12.38, **37** (29), 32-34.—In this region the glacial drift covering the outcrops may be as much as 300 ft. thick. Pre-glacial erosion has usually removed the Marshall Series, and wells pass directly into the Coldwater Shales. About 400 ft. below the top of this series is the Coldwater Lime, which varies in thickness from 30 to 40 ft. Underlying this is the "red rock," consisting of an argillaceous limestone series, and this is used as one of the main marker horizons. Between the "red rock" and the Antrim Shales are the Ellsworth Shales. The base of the Antrim series may contain a bed of crystalline dolomite which overlies the Traverse Shale. It is from a dolomitic limestone 60 ft. below the top of the Traverse that production is obtained. In the Salem field a second zone of fossiliferous limestone has also given production. The Dundee Series is not always present, and the Traverse may rest directly on the Monroe formation.

The Salem and Dorr fields are located on the same north-west to south-east structure, and it is possible that further discoveries will be made on this structural line. Other pools are situated on similar sharp anticlines, the flanks of which dip steeply. There appear to be two distinct structural trend lines in the region, one north-east to south-west and the other north-west to south-east, and it is thought that the basement is faulted in a north-west to south-east direction and has influenced later deposition.

Away from the crests of the structures the porosity of the Traverse Limestone decreases rapidly. This is possibly due to solution effects during the time when the higher parts of the structures were close to the surface of the sea and formed shoals or islands. J. A. G.

90. Geologic Aspects of the Forest City Basin. W. G. Osborne. *Oil Gas J.*, 5.1.39, **37** (34), 12-13, 22.—Throughout the whole of this area there is a series of north-south anticlines and domes which may be due to crumpling in the soft Pennsylvanian series rather than to deep-seated folding. It is also possible that the surface structures may mask the more important folds of greater structural depth. The author points out that probably the most favourable structures for oil accumulation are those overlying folds in the original basement rocks.

The sedimentary cover varies from 3000 to 4000 ft. in thickness, and the principal oil horizons should be discovered below the Pennsylvanian.

On a map included in the paper the author has marked several zones of varying importance, and a brief discussion of their possibilities follows. J. A. G.

91. Economics and Geology of the Rocky Mountain Area. L. C. Uren. *World Petrol.*, August 1938, 9 (8), 34.—From a geological point of view the Rocky Mountain area includes part of Southern Alberta, but should not include South-East New Mexico. However, the author considers the latter district in the paper, and excludes the Alberta area because it is not in the United States.

Oil and gas accumulations are definitely related to the sedimentary basins which contain suitable structures. Nine of these basins are listed, and according to H. F. Davies, oil formation was contemporaneous with deposition, the oil remaining in the central parts of the basins until folding movements took place in late Cretaceous times.

Oil is found in rocks ranging in age from Carboniferous to Tertiary, including seven main horizons. The Muddy, Dakota and Lakota Sands series at the base of the Upper Cretaceous has proved to be the best producing horizon in the area. The Wall Creek Sandstone (Upper Cretaceous) is also an important producer, and is responsible for much of the production at Salt Creek, the biggest field in the region.

After a brief summary of the individual fields, the author points out that any estimate of reserves is necessarily very approximate, but in 1937 they were computed at 817 million brls.

The paper also includes a detailed summary of the refining, storage, transportation and marketing methods.

J. A. G.

92. Progress in the Exploitation of Petroliferous Beds in Illinois. Anon. *Rev. Pétrolif.*, 18.11.38 (812), 1494.—Prior to 1936 the exploitation of oil in Illinois was not extensive. However, various discoveries were made in the southern part of this state, and the finding of deeper horizons, especially in the calcareous McClocky formation, in 1937 revealed the importance of these new fields. During 1938 the daily production has reached 100,000 brls., of which only 13,000 come from the old fields. In Illinois there has been more drilling in the past year than in Oklahoma or Kansas. Geophysical work has also been carried on actively. The fields are widely separated, and it is not unreasonable to suppose that the intervening area may hold good possibilities with regard to oil discovery.

G. D. H.

93. Oil Search in Papua and New Guinea. Anon. *Petrol. Times*, 10.12.38, 40, 765.—An exploratory borehole sunk to 1124 ft. showed traces of oil at several horizons, although there are no seepages within 7 ml. of the location. Strong gas-blows with a pressure of 400 lb./in.² were also encountered.

G. D. H.

94. Petroleum Prospects in Switzerland. J. Kopp. *Petrol. Times*, 3.12.38, 40, 721.—There are many indications of oil in Switzerland. Traces of oil are found in the Subjura zone, near Dardagny in the Canton of Geneva; on the Talenbach, near Chavornay; near Orbe; near Murgenthal; and on the Gouhard, near Aarau. Asphalt impregnations are seen in fissures near Ecelepeus, whilst a gas issue has long been active near Cuamay. The Val de Travers asphalt deposits have already yielded several million tons. In the Subalpine zone natural gas is found in the Ricken Tunnel, in the shafts of power-stations of the Waggital and of the Niederenbach at Engi, near Schwanden, as well as in the Rhineland, where recently gas was struck while drilling for water. Traces of oil have also been discovered on the Petersalp, on the Santis, in a well at Tuggen and in the Entlebuch. Oil and gas indications along the borders of the Swiss Alps continue into Bavaria and Austria, where in several places oil and gas accumulations have been exploited.

From his work on the Subjura zone Arm Heim concluded that the petroleum in the Oligocene Molasse is primary, but others disputed this, and Schardt traced the origin to deeper Triassic beds. As source-rocks of the Molasse oil the Meletta shales of the marine Rupelian seem likely. The Lower Tertiary supplies most of the Pechelbronn oil, whilst Rupelian beds contain important oil source-rocks in the areas bordering the Hungarian plains. However, there are evidences that the Rupelian was extensively eroded in the Subjura zone, but it may be present in the centre of the Swiss Molasse basin. No drilling has yet been undertaken on the favourable simple folds of the central area.

G. D. H.

95. Petroleum and Natural Gas-Fields of Poland—Boryslaw : Geology. Vol. 2, Pt. 1. K. Tolwinski. *Service Géol. des Karpates*, Bull. 22, 1934.—Boryslaw is situated on the north border of the Carpathians. Two important elements are involved in its structure: the thrust masses of the marginal Carpathians and the deep fold. The Miocene Salifère of the foreland extends for a considerable distance beneath these structures. The marginal thrust masses are divided into two tectonic units: the "skiba" of Orow and the border "skiba." (Skiba = a Polish word meaning the ridge of earth turned up by the plough.) The border skiba is developed in the form of a recumbent fold, overturned to the north, and with a Cretaceous core. It disappears gradually to the south beneath the skiba of Orow, and only where it is so covered does it contain important deposits of petroleum. The skiba of Orow covers an extensive area between the border skiba and that of Skole. To the east and west of Boryslaw it becomes more and more intensely folded. The culmination of the structure lies in the Boryslaw-Orow zone, whilst the Schodnica and Urycz folds are on the south flank. Relatively shallow deposits of petroleum are found in Cretaceous beds in secondary folds within the area of culmination. The true identity of the deep fold was for long a matter of speculation, but intensive surface mapping and drilling have shown it to be yet another "skiba," and it may therefore be called the Boryslaw skiba. As with the other skibas, the general form is that of a recumbent fold. In addition to the folding on the south flank, the structure is cut by a series of transverse faults, striking mainly south-west to north-east, and these probably extend to considerable distances on both sides of the skiba. The undulations of the fold in the direction of the longitudinal axis, as well as the secondary folding, influence decisively the distribution of oil, gas and water.

The stratigraphy of the area is dealt with in some detail in a second chapter. All formations from the Cretaceous to the Oligocene are included, whilst, in addition, there are the saliferous clays of the foreland which are Miocene in age.

The third chapter traces the development of the theories concerning the geological structure of the Boryslaw area. S. E. C.

96. Petroleum and Natural Gas-Fields of Poland—Boryslaw : Petroleum, Natural Gas and Ozokerite Deposits. Vol 2, Pt. 2. K. Tolwinski. *Service Géol. des Karpates*, Bull. 22, 1937.—The surface area of the Boryslaw field is some 15 sq. km. With the exception of shallow production from the border skiba and the skiba of Orow, the greater part of the production comes from the Boryslaw skiba. The various productive horizons in that structure are, from upper to lower, the Polanica beds, the Menelite shales, the Boryslaw sandstone, the Upper and Lower Eocene and the Jamna sandstone. Each of these is described in some detail. The production statistics for 1886-1936 and the production history of each of the horizons are given in a second chapter and in an appendix.

The importance and the commercial value of natural gas were not realized for a long time, and consequently there was enormous wastage. The annual production of gas per well in 1936 was approximately only one-tenth of that for 1918. Efforts to collect and use the gas were first made on a large scale about 1910, in consequence of the disastrous fires caused by free discharge. The first attempts to recover gasoline by compression were made in 1913, and the first adsorption plant came into operation in 1922. The percentage of methane in the gas varies between 50% and 80%, and there are small traces of helium.

The oil-field waters are divisible into three groups, both on a basis of hydrostatic pressure and on chemical analysis. The first group comprises the waters in the border skiba and the skiba of Orow. The geological conditions in the zone are complicated and the number of wells in production relatively few, so that it is very difficult to interpret the data. In contrast, the characteristics displayed by the waters in the second group—Polanica beds and Salifère formation—are considerably more regular. A very marked aqueous zone in the Polanica beds surrounds the culmination of the Boryslaw fold. There is no constant water zone in the Salifère formation, but the waters which occur sporadically have a very similar chemical composition to those in the Polanica beds. The third group of waters is subdivided into those in the Boryslaw sandstone, those in the Upper and Lower Eocene, and those in the Jamna sandstone. On account of the similar hydrostatic pressures and chemical compositions it appears that the waters in these three sub-groups must be in communication.

The Boryslaw crudes are divided into four groups. The first three groups come from the border skiba and the skiba of Orow, whilst the fourth includes all the crudes from the various productive horizons in the Boryslaw skiba. The uniformity of the waters and the crudes from the Boryslaw skiba points to a common origin. Edgewater encroachment is taking place in all horizons and at a greater rate in the lower.

Ozokerite occurs in longitudinal and transverse veins in the Salifère formation. The filling of these veins is frequently fragments of the neighbouring rocks embedded in a mass of wax, which is often accompanied by rock-salt. The thickness of the veins is very variable, and in some instances they have a lenticular shape. The fractures which the ozokerite has filled—due to escape of crude and loss of volatiles therefrom—are the final stage in the structural development of the area.

Details are given of the deeper prospecting wells in the Boryslaw area and of the history of the field. S. E. C.

97. Kirkuk, a Field Developed with High Technical Skill. Anon. *World Petrol.*, July 1938, 9 (7), 58-63, 124-126.—The field of Kirkuk lies about 200 miles north of Baghdad, and is situated on a normal anticline trending north-west to south-east. Production is from the Main Limestone of Middle and Lower Eocene age, which is unconformably overlain by the Fars Series of marls, clays, anhydrites, etc. The structure developed in the reservoir rock is a flat-topped dome about 60 miles long and varies from 1 to 2 miles in width. The flanks dip up to 50°, and are sometimes affected by faulting. Located on the main fold are three domes: Baba Gurgur, Avanah and Khurmala. Production has been proved in the first two for a distance of 50 miles, but only gas has so far been found in Khurmala.

Numerous wells have been drilled, and most of them are producers, although some are used to give information regarding edge-water movement. Drilling has been carried on gradually, and the rock pressures can be easily calculated, thus making it possible to weight the drilling mud correctly. The porosity of the producing limestone is very good, and wells as far as 30 miles from the nearest point of oil removal are affected. It was decided to commence production by removing 30 million brls. per year from an area roughly 10 miles long by 2 miles wide. About 105 million brls. of oil have so far been produced, and it has not yet been found necessary to alter the scheme.

A short review of the pipe-line and terminal points is also included in the paper.

J. A. G.

98. Natural Petroleums. C. Finaton. *Rev. Pétrolif.*, 16.12.38 (816), 1621-1624.—From 1859 onwards attempts to synthesize petroleum are numerous. Berthelot prepared acetylene, methane and ethylene. A few years later the organic theories of petroleum formation came into competition with inorganic theories, and in 1877 Radziszewski demonstrated the formation of CO₂ and a series of hydrocarbon compounds in the fermentation of cellulose. During the same year Berthelot and Mendeleieff investigated the action of water on metallic carbides. Eleven years later Engler's work on fish oils came to the fore. Sabatier and Senderens produced a whole series of hydrocarbons by catalytic hydrogenation, whilst in 1921-1922 Mailhe prepared a petroleum from vegetable oil.

The theories of Berthelot and Mendeleieff were taken up by some geologists, who accordingly proposed the existence of various substances deep in the earth, which on reacting with water would produce hydrocarbons; but various features associated with vulcanism refute these ideas. Hence geologists were drawn more and more to the organic theories. However, Engler and Mailhe required temperatures and materials (catalysts) which are not normal. Only the pressures are adequate, the temperatures calling for far too deep a burial.

The only reactions compatible with geological data are of the putrefactive types indicated by Radziszewski and Potonié. The natural laboratory of the sea has organic debris, aerobic and anaerobic bacteria, ionized compounds, catalysts of various types and prolonged time for reactions. The presence of optical activity in petroleum points to an organic origin. Recently cestrane, an animal product, has been found in petroleum. The occurrence of this unstable substance, which is only in small proportions in living matter, is remarkable. This hydrocarbon is also known in certain vegetables. The porphyrins, too, are found in natural bitumens and in almost all

petroleums (the oldest are without). Thus there is proof that petroleums have never been strongly heated. McConnell Sanders has observed all kinds of microscopic organic debris in petroleums. Hence their organic origin seems certain.

Fats are susceptible to hydrogenation, and bacteria can form hydrocarbons from cellulose. Methane and petroleum have been noted, amongst other things, in the mother liquors from the Stassfurt salts. The formation of paraffin hydrocarbons seems fairly straightforward, but the production of cyclic compounds presents more problems. Perhaps they were formed in areas of exceptional temperatures and pressures. In some of these areas geologists are said to have noted the lateral passage of carbonaceous to petroliferous beds, a fact which would be very significant.

G. D. H.

Drilling.

99. Humble Oil-Developing Means, Texas, Field in Orderly Manner. D. H. Stormont. *Oil Gas J.*, 10.11.38, 37 (26), 32.—The fact that the Means field, West Texas, is developed by one operator has enabled it to be worked in accordance with practices that make for the least amount of physical waste and result in the greatest ultimate recovery.

Only one drilling rig is being used. This is a steam-powered rig supplied by 3 125-h.p. boilers. The 122-ft. derrick is mounted on an 8-by-24-ft. steel substructure and the unitized draw works is driven by a 12-by-12 steam engine. Two pumps are used for mud fluid. Drill-pipe used is 4½ in.

No particular drilling problems are encountered, and in drilling the surface formations a rotating speed of 50–60 r.p.m. is used with the pump running slow and the weight on the bit varied, depending on the rate of penetration. When the salt and anhydrite sections are met, more weight is put on the bit, and the rotating speed is increased to about 125 r.p.m. In the lime sections the speed is again reduced to between 80 and 90 r.p.m., and the pump is run wide open.

As soon as the oil string is set, the 122-ft. derrick is skidded, a 94-ft. derrick is erected over the hole and the well is drilled in with a light portable rotary. By this means the drilling rig is kept in almost constant operation.

All wells are first tested naturally, after which they are acidized in either two or three stages.

Paraffin accumulation is the chief production problem.

L. V. W. C.

100. Pure Oil Co. Uses Tapered Seats for Casing Illinois Wells. H. Simons. *Oil Gas J.*, 24.11.38, 37 (28), 56.—The Pure Oil Co. are successfully running casing in wells in the Illinois basin area without the use of cement behind the pipe. This is being accomplished by the use of a tapered seat for the casing, which makes the landing of the string possible.

The method as used calls for the making of a 3-ft. seat tapered from 8¾ to 6¼ in., in which the casing rests. The taper is formed by special drag-type reamers with hard facing on the cutting edges. The Rosi Clare lime in the top of the Ste. Genevieve is the formation selected for the casing landing-point.

The mud is conditioned, starting at 2500 ft., until by the time the casing point is reached it has a weight of 10·2 lb. per gal. and 40 viscosity.

L. V. W. C.

101. Whipstocks are being Used in Deepening of California Field. T. P. Sanders. *Oil Gas J.*, 1.9.38, 37 (16), 30.—The most active locality in California is the 16-year-old Torrance field, where ninety wells have been completed during the past eight months in a newly discovered 4800-ft. sand.

Most wells in the Lomita area drifted to the north, which is up-dip, and for this reason directional drilling is often used in deepening, the object being to deflect the hole back to the south.

In the majority of deepening jobs all or part of the liner is side-tracked. Four different methods are used. A whipstock is being used for drilling out through the water string which obviates the use of cement as a base for the deflecting tool. After the whipstock is in place in the water string, a mill is used to drill the window.

It is necessary to use whipstocks frequently in directional drilling at Torrance, because the bit exhibits a strong tendency to remain vertical or to drift slightly north.

A dual-seal cementer is used to cement the upper stage. It consists of a 10-ft. joint of casing through which three holes have been drilled near the lower end. These holes are sealed with frangible phosphor-bronze discs which are dislodged by pressures greater than 2000 lb. A metal sleeve is welded around the joint, and at the top of the sleeve are ports closed during installation of the casing by thin strips of metal. A gun perforator is used to rupture the discs. Two cementing jobs can then be performed at the same time, and one shut-down period is thus saved. L. V. W. C.

102. Illinois Drilling Methods Reduce the Time Required on Well. H. Simms. *Oil Gas J.*, 15.9.38, 37 (18), 49.—The drilling campaign in the Southern Illinois basin requires rigs that can be rigged up with a minimum of time and expense, and yet be capable of drilling the hole in a few days, since the depth from which production is obtained does not exceed 3000 ft.

As a result most derricks are 96-ft. structures and some are even 85-ft. pumping derricks.

Generally the rigs are conveyed to the site on three trucks, exclusive of drill-pipe. Rigging up of rotary drilling tools requires only a short time.

Surface pipe is not set in many instances, and only thin mud is used after passing the 400-ft. mark, and as little mud as possible is used, owing to the cost of water.

L. V. W. C.

103. Difficulties of Killing Blow-Out in Coastal Louisiana Well. N. Williams. *Oil Gas J.*, 13.10.38, 37 (22), 44.—The Texas Co. No. 2 B State, in Vermillion Bay, off the coast of Iberia Parish, coastal Louisiana, was successfully killed, and this article is a résumé of the report prepared for the Louisiana Department of Conservation.

As the well was located in from 5 to 9 ft. of water 3 miles out in the bay, 16 miles by water from the base of operations and 27 miles from New Iberia, its blow-out and killing presented unusual problems. The well blew out on June 3rd from a depth of 9667 ft. while the 2 $\frac{7}{8}$ -in. drill-pipe was being removed.

Two 10-in., 6000-lb. test control valves were used in the Christmas tree, and the whole tree was assembled and flanged on a blow-out truck and loaded on a barge, which was towed to the well. Electric welding after the Christmas-tree connections had been stabbed into place resulted in small fires around the swage, and welding had to be discontinued. Unfortunately the whole tree was blown into the bay, resulting in the deaths of two men. A new Christmas tree was assembled, and with water being pumped in continuously, the well was eventually killed on July 17th.

L. V. W. C.

104. Advantages Gained by Use of Welded Casing in Oil Wells. E. W. P. Smith and R. L. Looney. *Oil Gas J.*, 1.9.38, 37 (16), 34.—Information is given as a guide to the welding of oil-well casing. The procedures outlined in this article are based on the experience of the past three or four years in California and Mid-Continent fields. The advantages of using welded casing and various factors to be considered are dealt with.

L. V. W. C.

105. New Rotary Drilling Equipment. Neubauer. *Oel u. Kohle*, 1938, 14, 757.—An account of several patented systems for transmitting and reversing the drive from diesel engines to drilling equipment.

P. G. H.

106. Characteristics, Methods of Combating and Economic Importance of Heaving Shales. Part 1. M. T. Halbouty and N. A. Kaldenbach. *Oil Wkly*, 24.10.38, 91 (7), 17.—This article deals with the geological and geographical distribution of heaving shales in the Texas-Louisiana Gulf Coast and their definition, classification and causes. A general outline has been adhered to in an attempt to present the many different phases of the heaving-shale problem.

L. V. W. C.

107. Characteristics, Methods of Combating and Economic Importance of Heaving Shales. Part 2. M. T. Halbouty and N. A. Kaldenbach. *Oil Wkly*, 31.10.38, 91 (8), 42.—The mechanical practices in combating heaving shales are described, and recommendations are made for additional studies on this important subject.

L. V. W. C.

108. Viscosity Characteristics of Clays in Connection with Drilling Muds. G. Broughton and R. S. Hand. *Petrol. Tech.*, 1 (4), Nov. 1938, A.I.M.M.E. Tech. Pub. No. 1002.—The clay suspensions were examined in MacMichael- and modified Bingham-type viscometers. The form of the time-torque curves for different setting times points to two distinct mechanisms causing resistance to shear, and even to two types of gel structure. Torque-r.p.m. curves, using asymptotic values from torque-time curves, were substantially straight lines. Alcohol and pyridine caused marked rises in yield value without greatly affecting the mobility, probably due to flocculation. Heating a bentonite suspension in a sealed tube at 100° C. for a week raised both the yield value and the viscosity at infinite shear. Barium hydroxide, but not sodium hydroxide, produced thixotropy in a bentonite suspension, indicating that it is not p_H value alone which controls the viscous properties of muds. The viscosities of different size fractions of kaolin and ball clay were measured, the values being higher for the smaller sizes, which also show yield values.

Alcohol diminished the stability of clay suspensions, presumably by reducing solvation. There appears to be no consistent connection between yield value and thixotropy. In aqueous ball-clay suspensions, yield value rises with rise in temperature. G. D. H.

109. Mud Technique in Iran. M. W. Strong. *Petrol. Tech.*, 1 (4), Nov. 1938, A.I.M.M.E. Techn. Pub. No. 1005.—Graphical routine records of all mud operations are kept showing: (1) gravity recorded on rig; (2) gravity recorded in daily laboratory samples; (3) Marsh funnel viscosity; (4) mud losses to formation; (5) pump pressures, average and maximum; (6) pumping speeds in deep wells; (7) remarks on general drilling conditions, additions to mud such as bentonite, brine, barite, etc., shows, formation changes and the like; (8) size of hole, size and type of drill-pipe and casing; (9) salinity of the mud where necessary. These charts are not necessary for normal wells, but for deep wells, or when drilling in difficult formations, such logs are of considerable value. Rotary flush sampling has been so developed that excellent well logs are obtainable for little time and expense, without resorting to coring for other than dip evidence or special research specimens, even under complicated tectonic conditions.

The main problems are the use of very heavy muds to combat high-pressure shows, the formation of suitable filter cakes and the conquering of lost circulation. Routine laboratory work calls for the chemical and physical examination of all mud materials contemplated for use, whilst research includes the investigation of sedimentation and allied processes, viscometry, the examination of surface tension and of sealing mechanisms. The use of oil-water emulsions for low-gravity drilling fluids is being tested.

The loss of pressure in a mud column due to gas-cutting is calculated.

G. D. H.

110. Drilling Patents. J. H. Howard. U.S.P. 2,138,006, 29.11.38. Rotary core drill.

J. H. Howard and P. C. Efromson. U.S.P. 2,138,007, 29.11.38. Reaming cutter mounting.

C. B. Wright. U.S.P. 2,138,719, 29.11.38. Blow-out preventer.

D. S. Anthony. U.S.P. 2,138,897, 6.12.38. Reamer.

R. G. Gates. U.S.P. 2,139,075, 6.12.38. Suction bailer.

R. G. Gates. U.S.P. 2,139,076, 6.12.38. Method of recovering or moving pipe by applying a strain to the pipe and suddenly allowing liquid in the well to enter an air-chamber to create a vertical surging of the liquid column.

A. Gray. U.S.P. 2,139,077, 6.12.38. Method of making drilling jars.

H. I. Davis. U.S.P. 2,139,444, 6.12.38. Device for recording inclination of a well by means of a recording liquid.

O. C. Snell. U.S.P. 2,139,525 and 2,139,526, 6.12.38. Blow-out preventer.

L. V. W. C.

Production.

111. Sun Oil Co. Completes Well with 6300 lb. Pressure on Gulf Coast. N. Williams. *Oil Gas J.*, 10.11.38, **37** (26), 41.—The pressure registered on the gauge of the Sun Oil Co. No. 1 Bayshore Farms in the Palacios field, Texas Gulf Coast, was more than 6300 lb., and this is claimed as the greatest pressure ever encountered.

The use of effective high-pressure well-control equipment and the exercise of care and precaution alone made control of this well possible.

Flowing through a $\frac{7}{8}$ -in. and a $\frac{1}{2}$ -in. choke in an auxiliary flow manifold 80 ft. from the well, and with pressure stepped down through a $\frac{1}{2}$ -in. choke at the well, the well is holding a working pressure of 5500 lb.

To facilitate the changing of chokes, the auxiliary flow manifold was provided.

The well blew out while the drill-pipe was being pulled and, out of control, it ignited. As the fire was about to be shot, a head of shale and mud from the well shut off the gas momentarily, snuffing out the blaze. The well was quickly capped and the flow-lines were connected up.

L. V. W. C.

112. Better Completions Reduce Gas-Oil Ratios in Goldsmith Field. D. H. Stormont. *Oil Gas J.*, 1.9.38, **37** (16), 45.—One of the most important recent developments in the Goldsmith field is the great strides made in reducing gas-oil ratios, brought about by more careful well-completion methods and the reworking of wells having too high a ratio.

The use of drilling time curves has become fairly widespread in this field, and many operators take advantage of the knowledge gained regarding a formation's characteristics to seat the casing point in a hard shell, if possible. Drilling time is also used to determine the total depths of wells, as well as to pick the point at which packers and liners should be installed to secure lower gas-oil ratios.

Much work has been done in perfecting the technique of acid treatment, and close studies are usually made of the reservoir formations before attempting to acidize.

L. V. W. C.

113. Factors which Influence Pumping Unit Selection. E. Kemler. *Oil Gas J.*, 18.11.38, **37** (27), 110.—The author shows that gear units for pumping should be applied on the basis of peak torque, and the data and theory presented indicate that: (1) the engine-drive service factors are not correct, and result in excessive penalties to properly equipped single-cylinder engines; (2) torsional vibrations can be set up in gear drives, and must be given some consideration in future design; (3) the use of maximum load in figuring peak torque will give units larger than necessary if based on calculated loads at high speeds; and (4) further consideration should be given to these facts by the Sub-Committee for rating of speed reducers.

The subject is considered mathematically, and numerous dynamometer cards are included.

L. V. W. C.

114. Cleaning Wells in West Texas by Means of Reverse Circulation. D. H. Stormont. *Oil Gas J.*, 3.11.38, **37** (25), 39.—Use of reverse circulation for cleaning wells, following their being shot, has become widespread in West Texas. In some fields the method is used entirely for cleaning out shot-holes.

This method of cleaning out takes advantage of the column of fluid having the highest velocity to carry out cuttings and debris from the hole.

A special kelly and swivel are used, and it is possible to obtain a fluid velocity sufficiently high to carry out cuttings and even large pieces of steel while using a low pressure and low-capacity pumps.

L. V. W. C.

115. Supplementary Barrel Checks Scale Trouble in Sucker Rods. J. P. O'Donnell. *Oil Gas J.*, 13.10.38, **37** (22), 61.—Since the introduction of air repressuring in the Middle District fields of Pennsylvania, corrosion of pumping equipment has become acute.

To overcome the deposition of scale, one operating company has developed a simple device which deflects the falling scale from the working barrel into a supplementary outside barrel, from which it can be removed.

The only departure from standard pumping practice when using this supplementary barrel is the drilling of four small-diameter holes in the top of the working barrel to permit ejection of the fluid.

When the well begins pumping, the rise of the liquid in the working barrel lifts the plug or scale deflector from its seat, first opening the ports in the working barrel, and then the top of the working barrel. When the power is shut off, the deflector drops back into the working barrel.

L. V. W. C.

116. Repressuring Gives Extended Life to Haynesville. J. C. Albright. *Oil Wkly*, 31.10.38, **91** (8), 31.—The Haynesville field has been given an extended production life by repressuring. The repressured area affects 279 of the 310 oil producers and utilizes thirty-two input wells. An average of 75 million cub. ft. of gas is required to operate this field monthly, with an average daily oil production of about 2300 brls. Since the casing was effectively set, very little workover jobs were necessary to permit the gas to be placed where it would do the most work.

The oil before repressuring had an A.P.I. gravity of from 34° to 34·5°, with the casing-head gas produced carrying up to as much as 10 gals. of gasoline per 1000 cub. ft. Due to solution of gas from repressuring, the gravity of the oil has been raised to an average of 35–35·5° A.P.I., whilst the gasoline content of the casing-head gas has fallen to an average of about 1·35 gals. per 1000 cub. ft.

Casing seats were found to be in excellent condition, and tubing was not used for injection, and packers were not used. Pressures run to as much as 275 lb. gauge, whilst in some cases the sand takes the proper volume with pressures as low as 18 lb.

When an injection well is found to show evidence of channelling, two methods are used. One is to place a higher back pressure on the pumping wells, and the other is to slow down the input volume of gas passing to the input well at fault.

Producing wells are usually tubed high enough so that the sand face is covered with oil at all times. By this clogging of outlet pores is eliminated, with the oil dissolving the paraffin more readily than if pumped dry.

L. V. W. C.

117. Pumping is Producing Method in Illinois. G. Triplett. *Oil Wkly*, 14.11.38, **91** (10), 54.—Pumping is the method of well production in the Illinois basin, and only in the McClosky lime fields and in the Salem pool are flowing wells found. The flowing life of lime wells in the McClosky field is only about 3 months, and preparations for pumping equipment are made at the time of completing wells. The life of the wells in the Salem field is not yet known, but long flowing-life is not anticipated, as the few pumping wells show no more than 400 cub. ft. of gas.

In all other sands the wells are put on the pump at the time of completion.

Individual well geared units outnumber central power installations.

The low gas output of sand-producing fields plus the desire to use gas for repressuring compels adoption of energy other than gas. Electricity and gasoline are the principal energies used.

L. V. W. C.

118. Solubility Relations and Volumetric Behaviour of Three Gravities of Crude and Associated Gases. J. E. Gosline and C. R. Dobson. *Oil Gas J.*, 18.11.38, **37** (27), 187; *Oil Wkly*, 21.11.38, **91** (11), 34.—A knowledge of the physical properties of reservoir flow is fundamental to a rational treatment of many problems in production engineering.

The results of three mixtures from flowing wells are given, showing such relationships as gas-oil ratio against specific gravity of the gas, the effect of crude gravity on gas gravity for various gas-oil ratios, relationship between gas-oil ratio and factor for conversion of formation volume to specific volume, etc.

The procedure for sampling and experimenting is explained and tables of experimental results are included.

L. V. W. C.

119. Oklahoma City Dolomite Wells Show Rapid Production Decline. H. Simms. *Oil Gas J.*, 13.10.38, **37** (22), 49.—Operators in the Simpson dolomite section of the Oklahoma City field are faced by two problems: a rapid decline and an ever-increasing encroachment of salt water.

Production is from the dolomite found from 6380 to 6450 ft., the pay being about 125 ft. thick. Production starts to decrease shortly after completion, and water

appears in ever-increasing amounts as long as the wells flow naturally. The water frequently reaches 60% of the total fluid from the well, but installation of pumps generally reduces the quantity of water, as time is then allowed for partial gravitational separation of the fluid; production, however, still declines.

Production in this field is uncurtailed because of the water saturation and the small amount of oil involved.

L. V. W. C.

120. Well Potentials are Being Determined by Use of Sound-Waves. L. G. E. Bignell. *Oil Gas J.*, 1.9.38, 37 (16), 49.—The new method for determining fluid levels in oil wells is by means of "echoes." By producing the well at varying rates and noting the corresponding fluid levels, it is possible to arrive at the capacity of some wells to produce.

The apparatus is known as an "Echo-Meter," and is electrically operated.

A description of the apparatus is given, as well as some practical results.

L. V. W. C.

121. Surface and Interfacial Tensions of Oil-Water Systems in Texas Oil Sands. H. K. Livingston. *Petrol. Tech.*, 1 (4), Nov. 1938, A.I.M.M.E. Tech. Pub. No. 1001.—The surface tensions of the oils and waters were measured with a Cenco-du Nouy simplified tensiometer, whilst the interfacial tensions were measured in the Bartell-type apparatus. A study of 40 samples showed a mean value for the surface tension of the oils of 30.4 dynes/cm., with a variation range of only $\pm 11\%$. The surface-tension values of the waters ranged 76 to 49 dynes/cm., with a mean of 66 dynes/cm. The fact that only five waters showed values greater than pure water, although inorganic mineral salts raise the surface tension, points to the presence of surface-active organic compounds, probably dissolved from the oil. The standard deviation of interfacial tension of naturally occurring oil-water systems was 7.3 dynes/cm. from an average value of 20.4 dynes/cm., and neither surface nor interfacial tensions could be correlated with geological age. Antonow's rule cannot be applied to these systems, the interfacial tension instead tending to vary directly as the surface tension of the crude. In general, the surface tension was higher for the denser and more viscous crudes. The temperature coefficient for interfacial tension was about -0.2 dyne/cm./° F. below 100° F., and -0.1 dyne/cm./° F. from 100 to 130° F. The interfacial tension was generally higher for the more paraffinic crudes. These results are of value in connection with water encroachment, water flooding, accidental water-flooding and emulsions.

G. D. H.

122. Flow of Oil-Water Mixtures Through Unconsolidated Sands. M. C. Leverett. *Petrol. Tech.*, 1 (4), Nov. 1938, A.I.M.M.E. Tech. Pub. No. 1003.—The flow of oil-water mixtures through unconsolidated sands was examined for cases in which the physical properties of the liquids, their proportions, the pressure gradients and the types of sand were varied. The concentration of oil within the sand was measured electrically. It was concluded that the relative permeability of an unconsolidated sand to oil-water mixtures is substantially independent of the viscosity of either liquid, but is related to the pore-size distribution, and to the displacement pressure, pressure gradient and water saturation which characterize the process. The effects of these variables are in the direction to be predicted from a knowledge of the Jamin effect. The most probable mechanism of flow appears to comprise segregation of most of the flowing oil in the larger channels between the grains, each channel being substantially full of oil, except at very low oil concentrations when the oil may move as discrete droplets. In either case some oil is thought to be stationary within the sand, whilst the water flows through channels not occupied by oil and, in the present case, as a continuous film around each sand grain.

The data presented substantiate the proposal that the relative permeability of a particular sand to either phase is a function of the water saturation and the dimensionless group

$\frac{\pi N}{PD}$ only.

G. D. H.

123. Interfacial Tension Between Water and Oil Under Reservoir Conditions. C. R. Hocott. *Petrol. Tech.*, 1 (4), Nov. 1938, A.I.M.M.E. Techn. Pub. 1006.—The drop-weight method was used for determining the oil-water interfacial tension in a closed

system in which the pressure and the gas content of the oil could be varied. The oil density was also measured within the closed system. Only sweet crudes were examined, and the interfacial tensions were all of about the same magnitude. The quantity of gas in solution affected the interfacial tension, which increased with the amount of dissolved gas, and consequently with the pressure required to keep that gas in solution. The effect of increasing the pressure on an oil containing a constant amount of dissolved gas was to decrease the interfacial tension slightly. A rough trend existed between interfacial tension and the density of the oil, the higher-gravity oils having lower interfacial tension against water. The surface tension of water against gas is greatly influenced by the pressure of the gas, ranging from values near that of pure water against air at atmospheric pressure, to values approaching the interfacial tension of water against crude oils at pressures in the neighbourhood of 3000-4000 lb./in.².

G. D. H.

124. Production Patents. G. A. Macready. U.S.P. 2,137,296, 22.11.38. Well fluid sampler.

D. W. Hoferer and E. T. Adams. U.S.P. 2,137,402, 22.11.38. Pump-valve construction.

D. W. Hoferer. U.S.P. 2,137,403, 22.11.38. Well pump.

T. E. Bryan. U.S.P. 2,137,441, 22.11.38. Gas lift valve.

H. Waller. U.S.P. 2,137,599, 22.11.38. Oil-well line-control device comprising a casing having a port in the side, a barrel and means for attaching it to the upper end of the well, a slide valve for closing the upper end of the casing and a lever for operating the valve, and a valve to close the port. Means is provided for opening the port valve as the slide valve closes.

J. D. Nixon. U.S.P. 2,137,853, 22.11.38. A polish rod device, including a guide sleeve, a socket on the end of the guide sleeve, a rotatable head connected to the socket and gripping means within the head for securing the device on to the polish rod. Packing is arranged to be deformed by the gripping means for forming a fluid-tight seal between the sleeve and the rod.

H. Salvatori. U.S.P. 2,137,985, 22.11.38. Method of logging a well by means of seismic waves.

W. E. Bendeler. U.S.P. 2,137,997, 29.11.38. Device for sealing between two telescopic related strings.

J. E. Hall. U.S.P. 2,138,002, 29.11.38. Well pump.

A. Hollander. U.S.P. 2,138,032, 29.11.38. Semi-submersible motor well-pump.

A. L. Armentrout and E. B. Hall. U.S.P. 2,138,057, 29.11.38. A method of testing the formation by positioning a perforated liner in the well carrying a drillable packing means lowered on a test string. The test fluid is allowed to enter the liner, and is removed, as is the string. The packing means is left in the well, and is then drilled up, leaving the well clear for further operations.

E. P. Halliburton. U.S.P. 2,138,156, 29.11.38. Well packer.

E. P. Halliburton. U.S.P. 2,138,157, 29.11.38. Removable packer for wells.

J. Grant. U.S.P. 2,138,375, 29.11.38. Well cleaning apparatus.

C. C. Brown. U.S.P. 2,138,569, 29.11.38. Well packer.

L. Spencer. U.S.P. 2,139,101, 6.12.38. Hydraulic pumping-jack.

W. T. Wells. U.S.P. 2,139,104, 6.12.38. Pressure-equalizing and surge-relief device for gun perforators.

W. T. Wells. U.S.P. 2,139,105, 6.12.38. Well fluid-pressure gauge.

W. H. Stigall. U.S.P. 2,139,319, 6.12.38. Tubing bleeder and jar.

G. Potapenko. U.S.P. 2,139,460, 6.12.38. Method of determining the presence of a substance having a selective effect with respect to frequency of an imposed electric field.

W. B. Lerch and T. M. White. U.S.P. 2,139,595, 6.12.38. Method of cleaning wells and well strings of deposits of paraffin wax, which consists of introducing into a well a water-miscible liquid wax solvent in the form of a column of liquid hydrocarbons, containing a small portion of modified fatty acid saponifying agent and flake naphthalene, and causing the column to be reciprocated up and down to dissolve the paraffin wax.

L. V. W. C.

Transport and Storage.

125. Calculation of Venting Requirements for Low-Pressure Oil Storage-Tanks. L. Bosa. *Oil Gas J.*, 18.11.38, **37** (27), 196; *Oil Wkly*, 21.11.38, **91** (11), 19.—A tentative standard method for the calculation of venting requirements is outlined. The application of this method to specific cases is in reasonable agreement with available experimental data and with current practice.

Since tank breathing is due to variations of the thermal conditions inside the tank, or to volumetric displacement due to the operations of filling and emptying, pressure or vacuum thermal breathing and pumping-in or pumping-out breathing are considered in this method.

A nomographic chart for the calculation of the venting requirements for low-pressure oil storage-tanks is included.

L. V. W. C.

126. Evaporation Losses of Petroleum Oils from Steel Tanks. C. C. Ashley. *Oil Gas J.*, 18.11.38, **37** (27) 170; *Oil Wkly*, 21.11.38, **91** (11), 13.—The investigation was undertaken in an effort so to correlate and compare all available tank evaporation-loss data that charts might be prepared to assist in making predictions of expected evaporation losses under given conditions, and in deciding on desirable roof construction or tank-venting facilities. More than thirty separate articles and reports covering over 200 individual tests have been reviewed. The original data were secured by various test methods. Owing to leakage losses, quantitative methods based on measurement of (a) vapours lost, or (b) changes in oil volume are only to be recommended as a check on other more accurate methods. Of the qualitative tests determined by change of (c) distillation characteristics, (d) composition of oil with respect to the most volatile fractions, (e) vapour pressure of the oil, the last-mentioned is regarded as the most accurate. The method of Chenicek and Whitman modified with respect to sampling method is considered the best available for the purpose.

The basic factor utilized in this work is the vapour pressure of oil at the average temperature of the tank, as it is considered that for all practical purposes the correction to the oil temperature eliminates from consideration all variables of weather, tank size and painting. This view is supported by a graph showing relation of atmospheric temperature to temperature of standing oil in steel storage-tanks based on data from tanks of various construction, capacity and degrees of filling, in tests of 5-62 days' duration. From the test data available, oil temperatures were calculated, where necessary. A correlation between Chenicek and Whitman vapour pressure at 75° F. and Reid vapour pressure at 100° F. was established. Curves are plotted to show: (1) evaporation loss from standing for full cone-roof tanks with and without vent-valves and tanks with pan-type floating roofs; (2) evaporation loss from cone roof-tanks due to pumping in; the Reid vapour pressure at 100° F. and average temperature of oil in the tank being related to evaporation loss in gals. per sq. ft. of oil surface per month in the first case, and to loss expressed as percentage of innage volume in the second. Breathing losses from breather roof-tanks standing full were negligible, and data for other types of roof not adequate. In plotting the curves of the first series, preference was given to tests by vapour-pressure methods over tests by gauging. Insufficient data were available to plot curves desired for variation of evaporation loss from cone roof-tanks with (3) degree of filling, (4) increase of vent-valve operating pressure. The data on which the report is based are derived from oils of relatively low Reid vapour pressure, and will probably not apply to oils of

high vapour pressure, owing to latent heat of evaporation effects if tank temperatures reach the boiling point of the product. The vent-valve pressures were of the order of 1-1½ in. of water, but appreciable reductions in evaporation losses are reported where higher tank pressures are employed. A suggested procedure for compilation of data and testing is given with a view to standardization and the subsequent availability of more accurate data upon which to build.

R. A. E.

Crude Petroleum.

127. Eastside Coalinga Field. California Crude Oil. Anon. *Petrol. Engr.*, Nov. 1938, 10 (2), 76.—A Bureau of Mines analysis of a sample of crude oil from Eastside Coalinga field, Fresno County, California indicates that it is an intermediate base oil with a gravity of 33.4° A.P.I. and a sulphur content of 0.22%. Yields on distillation are: light gasoline 7.0%, total gasoline and naphtha 26.9%, kerosine distillate 4.4%, gas oil 22.6%, non-visc. lub. dist. 11.3%, med. visc. lub. dist. 7.0%, visc. lub. dist. 1.9% and residuum 23.0%.

C. L. G.

128. Hydrocarbons in the Lubricant Fraction of Petroleum. F. D. Rossini. *Oil Gas J.*, 18.11.38, 37 (27), 141.—In connection with A.P.I. Research Project 6 a 10% fraction from crude oil from Brett No. 6 well in Ponca City, Oklahoma, has been separated into four broad fractions by solvent extraction and silica gel treatment. The fractions are: "wax" portion (35%), "asphaltic" portion (8%), "extract" portion (22%) and "water-white" oil portion (35%). The physical properties of the last two fractions are shown, and they were further fractionated exhaustively by distillation and extraction. Substantially constant-boiling fractions were then subjected to extraction. Each of the final "homogeneous" fractions was of the order of 15 gms., representing 1/40,000th part of the original crude, and consisted of compounds of substantially similar size and type. They appear to be nearer to pure compounds than any material, except *n*-paraffins, hitherto separated from the lubricating fraction of any crude. The "water-white" fraction was found to be substantially pure hydrocarbon material, and contained no sulphur, nitrogen or oxygen in significant amounts, whereas fractions from the "extract" portion contained about 0.9% sulphur, 0.1% nitrogen and 0.5% oxygen. The distribution of these elements in the fractions is discussed. On all important final fractions, ten properties were determined, and from some of these molecular formulæ, specific refractions, specific dispersions and kinematic viscosity indices were calculated. Certain fractions were subjected to hydrogenation and subsequently analysed to assist in determining composition of the fractions.

An illustration is given of the actual procedure followed in deducing the general identity of one of the final "homogeneous" fractions. Some of the correlation charts showing specific refraction, specific dispersion and aniline point plotted against number of carbon atoms per mol. are presented. Further charts, concerning the extract fractions, relate percentage extracted with methyl cyanide to b. pts. at 1 mm. pressure, kinematic viscosity at 100° F. and the kinematic viscosity index of one of the substantially constant-boiling fractions both before and after hydrogenation. Conclusions regarding the particular oil fraction tested are that: (1) about 43-51% of the lubricating fraction is composed of molecules with 1, 2 or 3 naphthene rings, together with appropriate paraffin side-chains; (2) about 8% as in (1), but with 1 aromatic ring; (3) about 8.1% with 2 naphthene rings and 2 aromatic rings (condensed), together with appropriate paraffin side-chains; (4) about 6.6% with 1 naphthene ring and 3 aromatic rings (condensed) with appropriate paraffin chains; (5) about 18-26% *n*-(straight chain) paraffins plus possibly some *iso*-paraffins (branched-chain); (6) about 8% of "asphaltic" constituents. Division of these types amongst the four fractions is discussed and shown in tabular form. An effort to separate pure hydrocarbons from a large quantity of a dewaxed lubricant fraction of Mid-Continent crude is under way, and further work on the "wax" and "asphaltic" portion of the crude discussed in the present report is desirable. The insight obtained into the composition of oil will assist in the manufacture of oils from crude for special purposes and in the production of synthetic lubricating and other oils.

R. A. E.

129. Chemical Constitution of the Extracts Portion of the Lubricants Fraction from a Mid-Continent Petroleum. B. J. Mair, C. B. Willingham, and A. J. Streiff. *Industr. Engng Chem.*, 1938, **30** (11), 1256.—A correlation is made of the properties of 179 fractions of oil prepared by extensive distillation and extraction of the extract portion of the lubricants fraction from a Mid-Continent petroleum. From a critical survey of the data the following conclusions are reached :—

(1) About 60% of the material consists of naphthenes with one to three rings per molecule;

(2) About 15% of the material consists of molecules with one aromatic ring and from one to three naphthenic rings per molecule, and with this material is associated a small amount of sulphur and oxygen compounds;

(3) About 14% of the material consists of molecules with two aromatic rings (linked through two common carbon atoms) and about two naphthenic rings per molecule, and with this material is associated a small amount of oxygen and sulphur compounds;

(4) About 11% of the material consists of molecules with more than two aromatic rings and one or two naphthenic rings per molecule, and with this material are associated some sulphur, nitrogen and oxygen compounds.

J. W. H.

Gas.

130. Patents on Gas. G. W. Johnson. E.P. 495,129, 8.11.38. Improved apparatus for the removal of foreign gases from circulation in catalytic pressure reactions.

Usines de Melles. E.P. 495,304, 10.11.38. Separation of olefines from gaseous mixtures by means of a preferential solvent for olefines, in a plurality of stages.

Standard Oil Development Co. E.P. 496,159, 25.11.38. Method of controlling temperatures in exothermic catalytic gas reactions. W. S. E. C.

Cracking.

131. Cracking of Kerosine in the Presence of Catalysts. C. Otin and S. Savencu. *Petrol. Z.*, 1938, **34** (46), 1.—A kerosine was heated under 1, 10 and 20 atm. pressure respectively at different temperatures for varying periods of time, both with and without catalysts (metals, oxides, chlorides, sulphates, etc.). The formation of gasoline was checked by specific gravity and distillation tests. In absence of catalysts, at atmospheric pressure (170° C.) and at 10 and 20 atm. (150–240° C.), no significant production of gasoline occurred on heating for 2–10 hrs.

In the experiments which employed catalysts the heating period was 2 hrs. in every case. Metals (K, Na, Ca, Zn, Al, Mg) and oxides (MgO, CaO, ZnO) had only a very small effect at 10 and 20 atm. The best yield of gasoline (6%) was obtained with MgO. Of the salts, AlCl₃ was by far the most effective in promoting cracking. At 250° C. (20 atm.) 31% gasoline was produced. The effect of time on the catalytic effect of AlCl₃ was studied separately. After 10 hrs. at 20 atm., 55% gasoline was obtained.

As a general result, it was concluded that in the absence of catalysts the influences of time and pressure were insignificant, but had an important effect when an efficient catalyst was used. P. G. H.

132. Corrosion-Resistant Steels in Cracking Plants. E. S. Dixon. *World Petrol.*, Annual Ref. Issue, 1938, **9** (11), 72.—The experience of a major oil company in the behaviour and economic value of corrosion-resistant steels in cracking service is outlined.

Carbon steel is usually preferred for the construction of vessels such as soaking drums and towers which can be protected by liners. For furnace tubes, bubble-tower angle iron tray supports, outside lines, cast valves and return bends, 4–6 Cr steel shows a service life of 3/20 times that of carbon steel, depending on the severity of corrosion, temperature, presence of protective coke deposits, etc. Plain Cr steel

tubes are liable to cold brittleness, but this disappears when heated, so that tubes should be warmed when knocking with coke-cleaning tools. The addition of Mo prevents this cold brittleness. The proper selection from the wide variety of alloy steels now available requires a complete knowledge of the particular conditions obtaining, so that designers sometimes build with carbon steel in order to determine where conditions are severest, etc. For relatively non-corrosive conditions 4-6 Cr-Mo steel is uneconomic and 2% Cr-Mo steel provides satisfactory service, but for severer conditions 4-6 Cr-Mo or 18-8 Cr-Ni steels are used. The air hardening during welding of Cr steel tubing in outside pressure lines can be prevented by annealing, for which an electric furnace has been developed. The Zeiss bore inspection telescope is used for the detection of defects in bores of hot-oil pumps. Liquid ends of hot-oil pumps have been reclaimed by re-machining oversize the ports and passages of the cylinders, filling with molten metal of 4-6 Cr composition and re-machining. The liner should be $5/6$ times more resistant to corrosion than the ordinary forged carbon cylinder. C. L. G.

133. Patents on Cracking. P. Ostergaard. U.S.P. 2,134,926, 1.11.38. Cracking of petroleum oils in which cracked products which are too low-boiling for use as commercial motor fuel are polymerized or cracked and then polymerized in a recycling operation.

W. E. Currie. U.S.P. 2,135,068, 1.11.38. Production of blended motor fuels of high anti-knock value and containing both polymerized and hydrogenated gasoline with or without cracked gasoline or absorption gasoline.

G. C. Peckham. U.S.P. 2,135,876, 8.11.38. Cracking of petroleum oils by direct contact with a suitable molten metal or inorganic salt heated to cracking temperature. W. S. E. C.

Hydrogenation.

134. Evolution of Processes for Preparing Gasoline by the Catalytic Reduction of Carbon Monoxide by Hydrogen. C. Berthelot. *Chim. et Ind.*, 1938, 40, 434-460.—The author considers that the commercial production of motor spirits by the direct hydrogenation of solid fuels is approaching its culminating point. On the other hand, the hydrogenation of liquid fuels, such as primary tars, oils from schists, petroleum residues, etc., has possibilities of great expansion.

The Fischer-Tropsch synthesis by which liquid fuels are prepared by the reduction of CO by H_2 is examined, and methods for preparing the gaseous mixture are discussed.

Instead of starting with the theoretical mixture $CO + 2H_2$ which is required by the Fischer process, it is possible to use ordinary water gas in a modification of the process suggested by Myddleton. This gives a lower yield of liquid products, but the resulting spirit has a higher octane number, due to less complete hydrogenation and the consequent higher content of olefines.

Methods for purifying the gases are discussed and the effect of various catalysts is described.

It is concluded that the best process, in general, is one combining the destructive hydrogenation of liquid products formed by distillation of solid fuels with the catalytic reduction of the water gas formed from the residual coke.

This can be carried out in one plant.

D. L. S.

135. Catalytic Hydrogenation Under Pressure of Albanian Crude and Products Obtained with Special Consideration to Gasolines. S. Doldi. *Chim. e Industria*, 1938, 16, 530-535.—The advantages of processing sulphur-rich asphaltic-base Albanian crude by hydrogenation are pointed out and the principles governing the process are outlined, with considerations on the influence of temperature, pressure and nature of catalyst. The various methods of processing the topped Albanian crude and the products obtained by catalytic hydrogenation under pressure are described. By the liquid phase process, with finely divided catalyst, medium and heavy stocks are obtained in great quantity, together with small amounts of naphtha. The selection of a particularly active catalytic agent is essential, since the latter, when finely divided in the

charging stock, prevents asphalt separation and ensures satisfactory hydrogenation. The characteristics of products obtained are described, the naphtha having an octane number of about 60, good stability, and very low sulphur, and is thus superior to the naphtha obtained by topping. Hydrogenation using a fixed catalyst is discussed, and difficulties encountered in this procedure are mentioned, pressures of about 600–700 atm. and suitable catalysts and catalyst blends are necessary. Hydrogenation in vapour phase is employed to convert medium oil into naphtha; this process is characterized by the high concentration of the catalyst used. The naphtha produced by this process requires no treatment other than an alkali wash. Two processes for vapour-phase hydrogenation are described, in one of which the product is automotive fuel and in the other aviation fuel. Using higher pressures and special catalysts, it has been possible to obtain from the Albanian medium stock an aviation naphtha of 77 octane number. P. G.

136. Destructive Hydrogenation of Roumanian Paraffin Wax. C. Candea and L. Sauciu. *Petrol. Z.*, 1938, **34** (50), 1.—A commercial, Roumanian paraffin wax, s. pt. 53–7° C., was simultaneously cracked and hydrogenated at different pressures, temperatures, and with variations in the free reaction space. Experiments were made with and without catalysts. When no catalyst was used, the products were unsaturated in character. In the presence of MoS_3 at 430° C., and with a small reaction space, marked cracking with hydrogenation occurred, but the light products so obtained contained considerable proportions of unsaturates. With a larger reaction space, however, the products were practically saturated (MoS_3 catalyst, 430° C.). P. G. H.

137. Synthesis of New High-Molecular-Weight Paraffins. H. Pichler. *Petrol. Z.*, 1938, **34** (38), 1.—In a study of the use of noble metals (Ru, Os, Pt, Ir, Rh, Pd) as catalysts for hydrogenation of carbon monoxide, it is shown that ruthenium in particular is far more effective than the more usual Fe, Co, or Ni in producing solid hydrocarbons. By a single passage of CO/H_2 mixture (1 : 2) at a speed of 1 litre/1 gm. Ru/1 hr. over the catalyst at 195° C. and 100 atm. pressure, 140–150 gms. solid and liquid hydrocarbons were obtained per normal cu. metre of "synthesis" gas. Working temperature and yields remained constant for 6 months in one particular experiment. Of the yield of hydrocarbons about 100 gms. per N cu. metre consisted of crude, white paraffin (m. pt. 118–119° C.); by treatment with solvents it was possible to isolate from the crude paraffin hitherto unknown solid paraffin hydrocarbons with m. pts. up to 132° C. and molecular weights 7000–9000. P. G. H.

138. Patents on Dehydrogenation. Universal Oil Products Co. E.P. 496,045, 22.11.38. Dehydrogenation of propane and butane for the production of aviation spirit.

C. Ellis. U.S.P. 2,137,275, 2.11.38. Process of reconstituting and dehydrogenating heavier hydrocarbons for the production of anti-knock motor spirit, i.e., conversion of heavy hydrocarbons with a limited formation of free hydrogen by bringing together the hydrocarbons and only an inert gas which does not enter into the reaction, and treating the mixture at temperatures above 350° C. and pressures of 300 atm. W. S. E. C.

Polymerization.

139. Gases from Skimming and Cracking Units are Polymerized to Form Gasoline. J. C. Albright. *Petrol. Engr.*, 1938, **10** (2), 78.—A description with flow sheet and analytical data is given of an Alco-Pure polymerization unit operating on Gyro-cracked gases at a refinery in Javella, La. The polymer plant handles 3,720,000 cu. ft. of cracked gas and 264,000 cu. ft. of recycle gas. The cracked gases are scrubbed to remove H_2S and compressed in two stages to 450 lb., the liquids removed being compressed to 370 lb., and both passed to the pyrolysis coil at about 90° F., where they are heated to about 1000° F. From there they pass to the polymerization coil, the reaction products being quenched to 440° F. and passed to a high and a low pressure

separator. The liquids are stabilized, giving liquids of desirable vapour pressure and an overhead gas which is liquefied for recycling through the polymer plant. The polymer liquid is pumped to the Gyro distillate feed and fractionated after being processed in clay chambers, the polymer bottoms being separated and passed to the Gyro-cracking unit feed-stock.

Analyses are given of the Gyro gasoline and of the mixture with polymer gasoline, the octane numbers being 70.2 and 72.3, respectively. C. L. G.

140. Phenol-Modified Indene Coumarone Resins. J. Rivkin and W. F. Sheehan. *Industr. Engng Chem.*, 1938, 30 (11), 1228.—The effects of varying the ratio of phenol to indene and indene-type unsaturateds in the polymerization reaction, taking place in the presence of an activated clay catalyst, is investigated and the properties of the resulting resins are discussed. J. W. H.

141. Patents on Polymerization. G. W. Johnson and I.G. Farbenindustrie A.-G. E.P. 495,121, 8.11.38. Conversion of olefines into liquid hydrocarbon products by polymerizing them in the presence of anhydrous aluminium chloride catalyst and perchlorethylene.

G. W. Johnson and I.G. Farbenindustrie A.-G. E.P. 495,184, 4.11.38. Catalytic polymerization of olefines into liquid hydrocarbons, using several separate reaction vessels.

W. W. Groves. E.P. 495,337, 11.11.38. Manufacture of resinous products by polymerization of a vinyl ester and crotonic acid.

Universal Oil Products Co. E.P. 495,477, 9.11.38. Conversion of paraffin hydrocarbons into olefines and then by polymerization into lubricating oils.

G. W. Johnson. E.P. 496,267, 24.11.38. Preparation of phosphoric acid catalyst.

L. F. Brooke. U.S.P. 2,135,793, 8.11.38. Preparation of phosphoric acid catalyst for polymerization processes.

J. W. Throckmorton. U.S.P. 2,135,923, 8.11.38. Production of high anti-knock motor fuel from gases rich in unsaturateds which have been liquefied and polymerized to convert the gases into gasoline.

W. N. Davis. U.S.P. 2,136,785, 15.11.38. Catalytic polymerization of normally gaseous olefines into liquid motor fuels, using a phosphoric acid film catalyst.

J. Hyman. U.S.P. 2,137,492, 22.11.38. Polymerization of unsaturated hydrocarbons containing diolefines, using granular, porous, synthetic complex silicates containing an Al ion.

F. E. Frey. U.S.P. 2,138,541, 29.11.38. Multi-stage polymerization process for converting normally gaseous hydrocarbons into motor fuels.

R. F. Ruthruff. Re. 20,931, 29.11.38 (cf. U.S.P. 2,017,325, 15.10.35). Improved polymerization process carried out at 850–1000° F. and under 500–2000 lb. pressure/sq. in. The fixed gases are separated from the products under high pressure and at relatively low temperatures and eliminated from the system. The remaining gases are separated from the liquid products in at least two subsequent stages at progressively lower pressures, and the gases rich in olefines separated from at least one stage and recycled for polymerization. The gases of low olefine content, separated in other stages from the system, are eliminated. W. S. E. C.

Refining and Refinery Plant.

142. Refining Gasoline with Solid Phosphoric Acid Catalyst. V. N. Ipatieff and B. B. Corson. *Industr. Engng Chem.*, 1938, 30 (11), 1316.—Experiments are described on the vapour-phase refining of gasoline with a solid phosphoric acid catalyst. The charging stock was untreated stabilized distillate obtained by cracking West Texas

crude oil. The catalyst was prepared from a mixture of diatomaceous earth (Sil-O-Cel) and commercial orthophosphoric acid, and the plastic mass was then heated to 250° C. for several hours, cooled, the solid cake broken up and screened to a convenient size. The following table exhibits results obtained in a typical experiment.

Refined gasoline :—

Barrels per ton of catalyst	5565
Barrels per ton per hour	10.3
Polymer, per cent. yield by volume	8.9

Hours on Test :—

Treating	541
Regenerating	114

Operating Temperature :—

	° F.	° C.
Preheater	520	271
Flashed vapour	440	227
Catalyst	350	177
Reboiler	435	224
Overhead vapour	255	124

Polymer formation can be decreased by using a shorter contact time and also by introduction of steam with the charging stock. Further, under the experimental conditions employed no cracking to gases occurred. Regeneration of the catalyst was accomplished *in situ* by air oxidation at 900° F. (482° C.). Fresh catalyst produced the equivalent of 1950 barrels of distillate per ton of catalyst, of good colour. The high-boiling polymer produced as a by-product has the characteristics of a drying oil, hardening to a resin on exposure to air. In addition, the phosphoric acid catalyst is shown to be superior to clay (Attapulugus) as a refining agent. H. E. T.

143. Factors in Doctor Sweetening. C. D. Lowry, C. G. Dryer, C. Wirth and R. E. Sutherland. *Industr. Engng Chem.*, 1938, **30** (11), 1275.—The use of excess sulphur in doctor sweetening has been found to influence adversely the effectiveness of oxidation inhibitors in sweetened gasoline. With adequate mixing of doctor solution, gasoline and sulphur at a temperature of not less than 85° C., followed by adequate settling, it is possible to produce a gasoline in which inhibitors will be consistently effective. The harmful effects of excess sulphur are apparently due to the formation of polysulphides, particularly those higher than trisulphides, which remain in the gasoline. J. W. H.

144. High Butane Recovery by High-Pressure Absorption. F. G. Noble. *Petrol. Engr*, Nov. 1938, **10** (2), 29.—A description with flow diagram, photographs, typical plant and data analyses is given of a high-pressure absorption process for butane extraction at Kilgore, Texas. The plant has a daily capacity of 8,250,000 cub. ft. and is designed to recover 95% of the butane. The compression plant operates in two stages, the first discharging gas at 38 lb. into four horizontal coolers whence condensate is removed, and the second discharging gas at 305 lb. pressure into three vertical high-stage gas coolers. Liquid is separated, and both streams are processed through the absorption distillation unit. The gas is delivered to a 64 ft., 24-tray absorber operating at the unusually high pressure of 300 lb. Four water intercoolers are used to restrict temperature rise of the absorption oil, whilst controls maintain a constant gas-oil ratio. Rich oil is vented into a tank at 130 lb. and the vapours pass to a 52-ft. 30-in.-diameter stripper still operating at 90 lb. pressure, whilst the oil passes through three stripper heat exchangers and a preheater into the stripper still.

In this unit the dissolved hydrocarbons in the feed, plus a small amount of absorption oil, are stripped overhead by steam entering the bottom of the tower where a high temperature is maintained by a reheater, in the return line of which the vapours from the rich oil vent tank are introduced. The overhead material from the stripper passes via a heat exchanger and two vertical stripper condensers into an accumulator, and thence into a vapour liquid separator, the gas being combined with the gas streams from the gasoline vent tank and still accumulator. After entering, the gas joins the liquid from the vapour liquid separator to form the total reabsorber feed. Vapours from the gasoline flash-tank entering the feed are obtained by flashing the high-stage

separator liquid from 300 to 130 lb. pressure. The flashed vapours pass through a back-pressure regulator directly to the reabsorber feed. The reabsorber is a 50-ft. by 30-in.-diameter tower operating at 85 lb.

The main still is a 52-ft. by 36-in.-diameter combination still and rectifier equipped with a reheater in the lower or stripper section. The still overhead enters two horizontal condensers where it meets the liquid from the gasoline flash-tank, both liquids then passing via a final sub-cooler into a still accumulator. The liquid product passes into a stabilizer raw-gasoline surge-tank, whilst the vapours are returned to the reabsorber. Lean oil from the still is combined with that from the stripper still, and after heat exchange passes to the lean-oil surge-tank for recirculation through the plant. Raw gasoline is charged to a 51-ft. by 36-in.-diameter stabilizer, the stabilized gasoline being heat exchanged and cooled, whilst the vapours are condensed and passed to an accumulator, the uncondensed vapour being utilized in the field residue system.

C. L. G.

145. Revivification Characteristics of Bauxite Used in Filtration. R. H. Hubbell and R. P. Ferguson. *Oil Gas J.*, 18.11.38, 37 (27), 135.—Previous work on bauxite has shown that in the percolation of residual stock, optimum results are obtained by operation at maximum practical temperatures of burning and filtration, whereas for decolorizing light-coloured distillate stocks minimum temperatures are best. A "solvent" effect, accompanied by a drop in oil viscosity, is exerted by new bauxite, but this is substantially lost after continued reburning. In the present work an analysis is made of the effect of revivification on decolorizing value and "solvent" effect of bauxite using plant-revivified samples from percolation of a variety of residual stocks, neutral and acid-treated distillates, dewaxed bright stocks and Pennsylvania petroleum stock. The conclusions are derived largely from operation of a plant in which bauxite is utilized through nine reburnings for distillate filtration, using low burning temperatures, followed by revivification at 1250–1300° F. for use in residual stock filtration. The relative efficiencies of new and revivified bauxite and new fuller's earth are compared. The results are presented in the form of graphs and of tables, which show properties of the oils before and after filtration as well as filtration and burning temperatures, absorbent density and percolation efficiencies on weight and volume bases. It is shown that whereas with fresh bauxite there is an appreciable reduction in specific gravity and Conradson coke value of the oil filtered, after several reburnings specific gravity and coke values are similar to those obtained by filtration through fuller's earth. Furthermore, as bauxite passes through several revivifications, the finished-oil cast improves until a point is reached at which the bloom is comparable to that imparted by fuller's earth. In general, the lower the numerical A.S.T.M. colour of finished oil from a given stock, the greater the reduction in efficiency of bauxite on revivification. After a certain number of burnings the efficiency reaches a practically constant figure for a given colour from each type of oil. The original efficiency of bauxite compared with fuller's earth varies according to the type and origin of stock, and so does the drop in efficiency on revivification. Bauxite shows to great advantage in the treatment of petrolatum, but drops rapidly in efficiency on revivification when used for treating Colombian distillate stock. Economic comparisons show bauxite to possess an advantage over fuller's earth in the decolorization of petrolatum stocks, residuals and of distillate stocks—provided that a balance is maintained, whereby bauxite, in its early stages, is segregated for distillate filtration. The use of bauxite for paraffinic distillates exclusively has been shown to be a borderline case. Selection of absorbent for a given service thus remains an individual problem for the solution of which the data presented provide a basis. R. A. E.

146. Curious Case of Increase in Decolorizing Power of a Mixture of Two Decolorizing Earths. II. E. Erdheim. *Przem. Naft.*, 25.12.38 (24), 656. (For Part I see Abstract No. 50).—It has been shown in Part I of this investigation that a mixture of two decolorizing earths "G" and "S," of low and high decolorizing powers respectively, had stronger decolorizing properties than would be expected from an arithmetical addition of their respective decolorizing powers, 3% of a mixture of "G" and "S" in different proportions being added to an oil 9/10° E₅₀.

In the second series of experiments this proportion was fixed at 25–75 "G," and the percentage amounts of the mixture added to the oil were being varied. Thus 0.5,

1.5, 3.0, 4.5% of the mixture were added to the oil. The results were again higher than those calculated from the decolorizing powers of the two earths. It was shown that these anomalies were not constant, but increased from 3.7 to 8.2% with increasing additions of the mixture.

A diagram is given illustrating the results obtained.

E. J. W.

147. Liquid-Liquid Extraction in the Separation of Petroleum Acids. H. G. Schutze, W. A. Quebedeaux and H. L. Lochte. *Industr. Engng Chem. (Anal.)*, 1938, **10** (12), 675-677.—Two rotary columns for counter-current extraction are described, one for the removal of the stronger acids first, the other for the weaker first. In the former case, between the upper and lower separating sections is the column proper, consisting of a glass tube within which a smaller rod is rotating at 200-500 r.p.m. The column is filled with a solution of the petroleum acids in petroleum ether to just below the upper section. 0.1N-KOH is added at the top at two drops per second, and 0.4N-H₂SO₄ near the bottom at such a rate that there is a slight excess of acid. The drops of the potassium salt solutions descend until they reach the acid inlet, where the free acids are liberated. Some dissolves in the descending water spiral, whilst most of the acids diffuse upwards, where interchange with descending potassium salt takes place, stronger acid replacing weaker. When 1 litre of KOH has passed, the aqueous layer is extracted with petroleum ether and added to the top of the column. The next litre of aqueous phase is extracted for cut 1, the next for cut 2, etc.

The other column is constructed on similar principles, and the results obtained on this separator with a complex petroleum acid mixture are quoted. Attention is also directed to the great advantages of fractional distribution, based on differences in K_a values and in structure, as a supplementary method to fractional distillation.

T. T. D.

148. Liquid Entrainment Separators. G. M. Kirkpatrick. *Industr. Engng Chem.*, 1938, **30** (11), 1207.—A non-mathematical analysis is given of the action of entrainment separators. The various factors affecting the performance are discussed in relation to combination centrifugal and contact surface and purely contact surface separators. Some figures are given for the capacities of the two types. P. D.

149. Study of Recovery of Oil from Spent Percolation Filters. C. M. Ridgway, L. M. Henderson and W. B. Ross. *Oil Gas J.*, 18.11.38, **37** (27), 154.—The investigation was limited to the fuller's-earth type of adsorbent and to Pennsylvania oils. The effect of viscosity on soakage (filling of voids between grains), draining and oil retention was determined by experimenting with neutral oils at 130° F. and bright stock at 200° F. Drainage decreases slightly with increased viscosity. The effects on washing efficiency of the variables volume, rate of addition and temperature of wash and type of naphtha used have been ascertained. Experience indicates that earth returned to the burner should not contain more than 2½% by weight of oil. An experiment carried out at 130° F. indicated that this was possible with neutral oils using reasonable amounts of wash, but excessive amounts of wash were required for the bright stock. A study was made of the soaking, draining and washing of Pennsylvanian cylinder stock solution filters in a plant equipped with filters of 35 tons nominal capacity of Florida fuller's earth, 16-30 mesh. Cleanliness of screen was found to be of prime importance in respect of drainage rate as well as total drainage. Factors affecting subsequent washing were examined, and attention was given to properties and filterability of oil recovered at various stages. Following the use of 11 brls. of naphtha per ton of clay, when oil content was 4.2% by weight of clay, steam was introduced to recover solvent and the oil content of the clay was reduced to 1.1% by weight. Effects of using recycle wash were also examined. The conclusions reached are summarized as follows:—

1. In plant filters the rate of draining and the total amount drained are dependent primarily on the condition of the screen.
2. In naphtha washing a low uniform washing rate is desirable.
3. The first portion of the washing naphtha should be applied at a temperature below that of the filter, and in such volume as is necessary to remove the filterable oil. The subsequent washing naphtha should be added at a temperature

above that of the filter for the purpose of dissolving an increased proportion of the tar.

4. A paraffinic naphtha of low vapour pressure and low end-point should be used in washing.

5. An appreciable increase in the viscosity of the original charge increases the amount of naphtha necessary to reduce the oil content of the filter to a specific magnitude.

6. The colour of the cylinder stock recovered in the clay wash is not indicative of its filterability.

7. The filterability of the oil contained in the washings is at present the only criterion that can be used in judging the fraction to be returned to the blend tank for reprocessing.

8. Clay-wash cylinder stock should not be blended with stocks of better filterability, inasmuch as this material decreases the filter yields to a much greater extent than its arithmetical proportion.

9. When recycle wash is used, extreme care must be taken to avoid introducing into the system material of poor filterability.

R. A. E.

150. Continuous Residuum Coking by Delayed Coking Process. R. J. Kiwocky. *Oil Gas J.*, 18.11.38, 37 (27), 131.—The "delayed coking" process developed by the Standard Oil Co. (Indiana) has been in commercial operation for several years, and has been applied to reduced crude and in a combination crude-running and cracking plant. The plant described was constructed in 1930, and has a capacity of 2860 brls. per day of Mid-Continent reduced crude of 19.0° A.P.I. gravity. Reduced crude at a temperature of 685° F. is pumped to the furnace, a typical radiant—and convection—section heater. Feed enters at the bottom of the convection section and passes upwards counter-current to the flow of gases. Parallel flow through two tubes is employed in the lower half and series flow in the upper half. The oil then enters the radiant section, in which only series flow is employed. The radiant section contains tubes on each side wall and on the front wall, and the hot oil passes in a U-shaped path through the lowest tubes on the three walls, then back over the reverse route in the second lowest tubes, and so forth. After passage through the wall tubes the oil flows through a single row of roof tubes, leaving the furnace normally at 910° F. Firing is controlled by the oil-exit temperatures. The residuum then enters a "circulating ring" flowing around in two directions from the point of entrance, the flow in each leg being regulated by manually operated valves and controlled by temperature. The two streams meet at the point of exit and enter the coking-drum, and in this way the undesirable deposition of coke at various points is avoided and diversion of the stream from one drum to another is facilitated. Three coking-drums are provided for continuous operation. Flow of oil is at a uniform rate, until the drum is filled with coke, the time required being determined by previous experience. The drums are of riveted construction and of special design, coke removal being effected by cable pulling followed by hand removal from walls. The method of fixing cables and the cycle of operations are fully described. Hot vapours from the coke drum pass to a fractionating tower, naphtha passing overhead and gas oil suitable for cracking stock withdrawn from the base. The length of runs, approximately 900–1000 hrs. of continuous operation, is generally limited by coking within the furnace roof tubes and sometimes in the upper wall tubes.

Yields by weight obtained from the particular residuum are approximately 22.4% gasoline of A.S.T.M. octane number 55, F. B. pt. 400° F., Reid vapour pressure 8 lb.; 69.1% of gas oil, 4.5% gas, 12.4% coke. The coke produced differs somewhat throughout the drum, being more dense and containing less volatile matter in the lower section. Principal conditions controlling coke hardness appear to be: (1) furnace outlet temperature, (2) time coke remains at elevated temperature, (3) efficiency of drum insulation, (4) characteristics of residuum. Conditions can thus be adjusted by several means to produce the hardest coke consistent with ease of removal from the drum.

R. A. E.

151. (Atlantic Refining.) Operation Data for Sludge Conversion Contact Acid Plant. W. B. Hart. *World Petrol.*, Annual Ref. Issue, 1938, 9 (11), 117.—The usual method of recovery of acid from petroleum refining sludges by separation of sludge and con-

centration of acid leaves the problem of disposal of the residual sludge. A method has now been developed for the decomposition of the sludge with heated coke, the purification of the gases, combustion to remove any residual hydrocarbons (any H_2S available being added at this stage), and the conversion of the SO_2 formed to sulphuric acid by the contact process. Acids of 80% H_2SO_4 up to 20% fuming acids are produced, 89–90% of the original acid in the sludge being converted, whilst with H_2S a 94% conversion is obtained. The plant described has a capacity of 65 short tons of 100% acid per 24-hour day, 50 tons from sludge and 15 tons from H_2S , or 50 tons of 98% H_2SO_4 and 15 tons of 20% fuming acid. As by-products 13 tons of dry granular coke and 1300 gals. of recovered oil are obtained.

The decomposer consists of a revolving kiln in which the heated coke and sludge are introduced. Separate charging systems for naphtha and for heavier sludges are advisable, as separation of acid is liable to take place when the two are mixed. The temperature of the outlet coke is controlled at 500–550° F. The vapours from the decomposer are drawn off through a flue to an overhead spray scrubber 5 ft. in diameter and 18½ ft. high, where coke dust is removed, and thence to the spray cooling tower, to condense as much water and oil as possible. They then pass to the oil separator, the outlet temperature of which is controlled at not more than 90–92° F., in order to limit the amount of hydrocarbon vapour. The remaining hydrocarbons are removed in a horizontal cylindrical furnace operating at 1400–1800° F., into which any H_2S available is admitted. The gases leaving the furnace at about 1500° F. contain about 12% SO_2 and are cooled by a water-spray. Thence the gas passes into the contact acid system, consisting of Cottrell mist precipitator, 60° C. acid dry tower, blower, converters and absorbers. Operating details, plant organization and typical analyses of sludge are given.

C. L. G.

152. Flexibility Characteristics of Recently Completed Manchester Refinery. Anon. *World Petrol.*, Annual Ref. Issue, 1938, 9 (11), 138.—Full details are given of the Manchester oil refinery plant at Manchester, which consists of a Foster Wheeler high-vacuum distillation unit of capacity 150,000 tons p.a., a combined solvent treating and dewaxing unit using benzol/ SO_2 with a capacity of 40,000 tons p.a., a de Laval acid-treating plant and a contact clay-treating and filtration plant with an annual capacity of 60,000 tons, storage tanks of approximately 30,000 tons capacity, boiler plant and complete auxiliary equipment.

The distillation unit can handle heavy crudes or residuum, and is designed to produce kerosine, gas oil, three lubricating-oil fractions and a residue. The use of benzol and SO_2 permits wide variation in the selectivity of the solvent extraction, whilst separation of the two components is easily obtained. A 25:75 benzol/ SO_2 mixture is usually employed, the relative positions of the raffinate and extract layers in the plant being ascertained by a neon-light system based on the different conductivity of the two liquids. The raffinate solution is diluted to give an 80:20 benzol/ SO_2 mixture and passed to the dewaxing filters, the cooling being obtained by the evaporation and compression of SO_2 in a special cooling system. The refinery programme visualizes the production of 20,000 tons of solvent-extracted lubricating, transformer and white oils, and up to 35,000 tons of acid-treated lubricating oil, a total of 60,000 tons of kerosine, gas oil, diesel and fuel oils, axle oil, dark cylinder stock and 10,000–20,000 tons of bitumen. As by-products of the solvent plant there will be a production of 3000–5000 tons of paraffin waxes, and possibly a small production of petroleum jelly if required.

C. L. G.

153. Houdry Processes for Catalytic Treating of Petroleum. E. Houdry, W. Burt, A. E. Pew and W. A. Peters. *World Petrol.*, Annual Ref. Issue, 1938, 9 (11), 68.—The processes described include: (1) catalytic cracking of crude or any fraction thereof (except gasoline) with or without the production of tar or heavy liquid products; (2) catalytic viscosity breaking; (3) catalytic treatment of gasoline from thermal or catalytic cracking; (3a) liquid phase catalytic treatment of aviation gasoline from catalytic cracking; (4) catalytic desulphurization of gas from cracking or topping operations; (5) catalytic polymerization in liquid phase of butenes; and (6) catalytic cracking of heavy gas oils or of residuums to lighter gas oils for furnace oil requirements.

1. In catalytic operations flexibility of design permits of a wide variety of stock being handled, including the direct cracking of the entire crude. In general, from

single pass operation on any stock is obtained a yield of 45% of gasoline of O.N. 77-81 for 437° E.P. gasoline, with high lead susceptibility and blending value. Catalytic gas oils are suitable for catalytic or thermal cracking. A typical flow sheet and details of yields from various stocks are given. A particular feature is the ability to produce 76-78 O.N. aviation gasoline, which can be brought to 87, 90 and 92 O.N. with 1.8, 3 or 4 mls. of tetraethyl lead per gallon, respectively. Seven million gallons are stated to have been made in 1937-38. A 100 O.N. (army method) blend marketed consisted of 25% *iso*-octane, 75% of Houdry aviation gasoline and 2.75 c.c. of lead. Typical properties of Houdry aviation gasolines are tabulated and also the properties and yields of products from the catalytic cracking of various gas oils. In the running of crude oils, non-residuum operation can be carried out by replacing the tar separator vaporizer by a director catalytic vaporizer in which the residual carbon is deposited on the catalyst and subsequently removed during regeneration by burning off. Yields from Mid-Continent crude bottoms, Lagumillas crude and West Texas (New Mexico) bottoms are tabulated, being 39.4-47.7% gasoline of O.N. 78.6-79.8, the remainder being gas oil without the production of heavy oils.

3. The catalytic treating operations described cover: (a) vapour-phase treatment of catalytic or thermal gasoline and (b) liquid-phase treatment of catalytic aviation gasoline.

The untreated catalytic gasoline is of low gum content (induction period of 10 hrs. or more), low sulphur, colours generally sufficiently good to enable dyeing, but will not pass doctor or corrosion test, although a caustic-soda wash enables it to pass the corrosion test. The treatment causes no loss of O.N., and yields a very low-gum, doctor sweet gasoline of high stability, low sulphur, excellent colour and odour. Operating costs vary from 2.5 to 4.5 cents per brl. The treatment consists of passing the vaporized gasoline at about 600° F. and 15-100 lb. pressure over moulded solid catalysts of acid-treated clays containing oxides of heavy metals, Mn and Ni.

4. Catalytic desulphurization is available for the almost complete removal of sulphur from refinery gases, the gas being heated to about 750° F. and passed over supported nickel for 30 mins. or more. Coke-oven gas containing 400 grains of H₂S and 45 of CS₂ was processed, removing 96.5% of CS₂ and reducing the H₂S to less than 1 grain per 100 cub. ft.

5. A selective liquid-phase catalytic polymerization process treats butenes yielding motor gasoline of A.P.I. gravity 57, O.N. 84-85 and blending value 136. Yields of 55-72% of unsaturates are given in one pass, the polymer being an excellent base stock for hydrogenation to *iso*-octane.

2 and 6. Yields are also given of the viscosity breaking of residuals from West Texas and mixed coastal and East Texas crudes, yielding 1 and 2% of naphtha, and 3 and 5% of gas oil, respectively.

Regeneration of the catalysts in all processes is necessary, the activity decreasing owing to carbon deposits. Operating cycles vary from 30 to 135 mins. in cracking fuels and gas oils, from 8 to 12 hrs. in vapour-phase treating operations and from 6 to 12 hrs. in the polymerization operation. The life of the catalyst is unlimited, no reduction in yield being experienced over 12 months' operation. The catalyst cost for a plant charging 15,000 brls. daily amounts to \$36,000.

The extreme flexibility of the operations and the higher yields of valuable products and the wide variety of stocks which can be treated are emphasized. C. L. G.

154. Cleaning Heat-Transfer Equipment. G. R. Hersam. *Petrol. Engr*, Nov. 1938, 10 (2), 71.—A discussion is presented of the main types of deposits formed in heat transfer equipment and of the more important cleaning methods and compounds in use. Deposits may be either oil residues—*e.g.*, sludge, carbon, salts, silt, etc.—or water and corrosion scales or rust, whilst slime and algæ may also be present. The deposits may be hard, porous or loose, the porous type being regarded as more serious owing to its lower conductivity. Drilling or rodding out is troublesome, and liable to cut thin walls of small heat-exchange equipment. Circulation of HCl to remove water and scale is liable to corrode equipment, but special inhibited acids are more efficient, and are not appreciably corrosive. Soft and loose deposits—*e.g.*, sludges and some oil residues—may be removed by blowing hot water or steam through the unit or by swabbing. Plastic plugs may be forced through the tubes, but are of no value for hard scales.

Modern methods consist of circulating specially selected cleaning compounds at nearly boiling point with violent agitation through the equipment, followed by flushing with steam or water. It may be preferable to remove tube bundles and immerse them in the solution. Completely plugged tubes must be rodded out first. It is recommended that there should be a permanent installation of cleaning equipment and that where possible spare sets of heat exchange, etc., equipment should be installed for use while one set is being cleaned.

C. L. G.

155. Hydraulic Decoking of Coke Chambers. W. F. Court. *Oil Gas J.*, 18.11.38, 37 (27), 179.—A new method of removing coke from coke chambers now in commercial use consists of cutting the coke by means of high impact-producing water-jets.

The unit described is in operation at Wood River Refinery of Shell Petroleum Corporation in conjunction with a combination cracking and coking unit processing 16,500 brls. a day of topped crude, gas oil and pressure distillate and producing 7000 brls. of 70 octane gasoline and 300 tons of coke per day. The four coke chambers are used in a 48-hr. cycle, only one at a time being on stream. Each chamber is filled within 10 ft. of the top, equivalent to approximately 150 tons of coke, and two chambers are cleaned every day. The average length of run on the coking section of the plant is 30 days. Hydraulic cutting nozzles are attached to special tools mounted at the end of a hollow stem, to which water is supplied from a high-pressure header through a steel-piping linkage made flexible by ball-bearing swivel joints. This stem is suspended from an overhead structure mounted above the chamber, and is lowered and raised by means of an air hoist.

The operation involves two steps: (1) Boring a hole 18 in. in diameter downwards through the coke, which is accomplished by means of a special boring tool, in two sections, to save excessive height in the overhead structure; (2) cutting and removing the coke from the chamber. When the boring tool emerges from the bottom manway, it is replaced with a part of the assembly of cutting nozzles. It is elevated into the chamber in order to make an enlarged opening in the bottom of the coke-bed. The complete cutting assembly is then attached and raised into the chamber. In the first cutting operation the nozzles are offset to give rotation, and the jet of water issues horizontally, penetrating to the chamber wall. The complete assembly has two nozzles pointing slightly upwards at an angle, and serves to cut the coke away from the wall. As the coke is cut into layers by the revolving jets, it breaks away and falls to the bottom of the chamber, and into cars. The water draining falls on to a concrete floor, and the fine coke is washed into trenches, accumulated in a pit and pumped into partly filled cars in which the coke-bed retains the fines. The rate of flow of water to the cutting nozzles is approximately 700 gals. per min. under a pressure of 900–1000 lb. at the entrance of the nozzles. Considerable research was necessary to discover the best type of nozzle and optimum pumping rates and pressures. As a result it is possible to remove completely 150 tons of coke from a chamber and have it ready for heading up again in less than 2 hrs. Coke so hard as to present difficulties in removal by the cable method is easily removed by the hydraulic method. Advantages claimed over the cable method are: (1) clean-out time halved; (2) saving in labour; (3) saving in cost of cables, etc; (4) elimination of cable-fastening wire from coke produced; (5) complete coke removal from chamber wall; (6) elimination of hazard, since no clean-out crew is required to enter the chamber at any time. The cost of maintenance of jetting nozzles is low, and tests have indicated that the cutting action has no effect on the steel walls of the vessels.

R. A. E.

156. Technical and Economical Aspects of the Thylox Process. W. Fitz. *Brennst.-Chemie*, 1.11.38, 19 (21), 397–402.—The development of the Thylox process operating in one stage with clear solutions, and yielding elementary sulphur, is described. The process is at present used in twenty-one plants with a total yearly output of almost 120 milliard cub. ft. of gas. Two Japanese Thylox plants are described, and the results obtained are given. One of these two plants is used for the purification of coal distillation gas diluted with water gas, whilst in the other plant blue water gas is freed from hydrogen sulphide. It is of special interest that the hydrogen sulphide content of blue water gas is reduced, solely through the application of the Thylox process, to 1.45 mgr. per cub. metre, which is far below the allowable maximum of 20 mgr. per cub. metre.

The economy of the Thylox process is discussed, based on data from various plants, and operating cost are plotted against hydrogen sulphide contents for varying gas output in the case of coke-oven gas and water gas, respectively. In the purification of great quantities of coke-oven gas in certain cases a net operating credit results. By virtue of its great economic and operating advantages, together with the efficient elimination of hydrogen sulphide, it is likely that the Thylox process will be introduced in Germany for the purification of water gas and of the gaseous hydrocarbons for oil synthesis.

L. R.

157. Alloy Steel Valves for Sub-Zero Temperatures. G. F. Scherer. *Industr. Engng Chem.*, 1938, 30 (11), 1220.—Although the tensile strength, yield point, modulus of elasticity and hardness change favourably with decrease in temperature, the shock resistance is affected adversely with a decline in temperature. High nickel-copper alloys and austenitic chrome-nickel-iron alloys are the best metals known to possess high impact resistance at sub-zero temperatures. Specifications are given for material suitable for working temperatures down to -150° F. at a pressure of 600 lb. per sq. in.

J. W. H.

158. Centrifugal Pumps for the Process Industries. H. E. LaBour. *Industr. Engng Chem.*, 1938, 30 (10), 1105.—The history and performance characteristics of centrifugal pumps are given briefly. There is available a wide range of materials from which pumps may be constructed, and these are considered in some detail in relation to the duty of the pump. Slight constructional modifications are advantageous in many cases, and these are also discussed.

P. D.

159. Plate and Frame Filter Press. P. Kriegel. *Industr. Engng Chem.*, 1938, 30 (11), 1211.—The advances in the design of filter presses to meet specific process requirements are reviewed. These advances include special corrosion-resisting materials of construction and gasketed plates and frames capable of operating at pressures up to 1000 lb. per sq. in. Typical installation and operating cost data are given for the recovery of filter cake from a chromium hydroxide slurry and for the clarification of cider.

J. W. H.

160. Patent on Refinery Plant. K. Koppers. U.S.P. 2,137,722, 22.11.38. Distillation apparatus for the distillation of high-boiling liquids such as coal-tar at decreased pressure.

W. S. E. C.

Chemistry and Physics of Petroleum.

161. Application of Catalysts in Organic Chemical Industry. G. Natta. *Chim. e Industria*, November 1938, 17, 719-724.—The development of catalysts in recent years is outlined and the principal applications in industry are examined. Particular attention is given to catalytic hydrogenation and dehydrogenation. The application of catalysts in the hydrogenation of mineral oils and residuals is mentioned, as well as the processes used: liquid phase with suspended catalyst and liquid phase with fixed catalyst for products boiling under 400° C. Interesting is the fact that by appropriately varying the operating conditions it is possible to obtain from certain raw materials either products mainly consisting of normal paraffin hydrocarbons, or branch-chain hydrocarbons, or naphthenic hydrocarbons. The hydrogenation of the double-bond hydrocarbons is at present very simple, being obtained at low temperatures, whilst the yields are good (the hydrogenation of the *iso*-octene to *iso*-octane takes place at normal temperature). The catalytic hydrogenation process, besides being used for preparation of glycerine from hydrocarbons, is employed in the synthetic rubber industry, where it represents one of the most important intermediate steps (transformation of the aldol in butylene glycol): this reaction, taking place at very low temperatures, necessitates a very active catalyst. The catalyst reactions of dehydrogenation are mentioned, and those employed for the synthesis of rubber (Buna S) for passing from butylene to butadiene, and from ethylbenzene to styrene, are pointed out. Also the dehydrogenation of alcohols has found application

in Italy—for instance, in the preparation of acetone from *isopropyl* alcohol. Good results have been obtained employing catalysts of the alumina and kaolin type for the hydration and dehydration reactions. From the first industrial process of this type synthesis of acetaldehyde from acetylene in presence of mercury oxide, it was found possible to prepare in this way *isopropyl* ether suitable for the preparation of aviation fuels. Serious difficulties have been encountered in effecting the catalytic reactions of oxidation, on account of the disadvantages resulting from the high reaction heat. In this connection are mentioned the oxidation of ammonia, of aldehyde into acetic acid, the catalytic oxidation of benzene to phenol, and the oxidation of aliphatic hydrocarbons to fatty acids. The condensating and polymerizing action of catalysts is then examined; the preparation of *iso*-octane from *iso*-butylene is mentioned, and the procedure for the preparation of synthetic rubber Buna S is illustrated. This process consists of different phases: hydration of acetylene and acetic acid, condensation of aldol, reduction of aldol to glycol, dehydration of glycol to butadiene and polymerization of butadiene. The various functions of the catalysts and their application in an extensive branch of industry are set forth. Some suggestions as to the inherent mechanism of these reactions and on the characteristics of the catalysts employed are given. Stable, resistant and very active catalysts must be available, and, owing to the high investment cost of equipment, it is necessary to reduce the volume of catalysts to the minimum in order to have maximum throughput. In plant operation, where catalysts are used under temperatures close to the melting point, it is necessary to use small amounts of inert substances, so-called promoters, which prevent undesirable re-crystallization. The active surface should be porous, but not to a too great extent—about 40–50% of the volume—since otherwise the mechanical resistance would be impaired; its action should be the surface absorption of the reacting gases. A. R.

162. Infra-Red Absorption Spectra of Some Naphthalene Hydrocarbons. P. Lambert and J. Lecomte. *Ann. Off. Combust. Liq.*, 1938, **13**, 111–126.—The authors have examined the infra-red absorption spectra of the following hydrocarbons:—

α - and β -benzyl-naphthalene, 1:4- and 1:8-dibenzyl-naphthalene, indene and *iso*-butylindene.

The results are given in the form of curves showing the amount of light transmitted, plotted against the wave-length. A fact which the measurements have shown is the importance of the region from 15 to 20 μ in the identification of hydrocarbons. The various maximum regions of absorption for the hydrocarbons examined are given in tabular form.

The paper discusses the effect of the constitution of the hydrocarbons on the type of spectrum obtained. W. E. J. B.

163. Fractionation of Mineral Oils by Gas-Saturated Propane. M. Godlewicz. *Petrol. Z.*, 13.7.38, **34** (28), 1–12.—This paper describes the use of gas solutions for fractionating heavy hydrocarbon mixtures. The mineral oil is dissolved in a solvent such as propane, which is then treated with a gas—*e.g.*, methane—under pressure. The gas dissolves and two liquid layers are formed. Stepwise fractionation can be obtained by using gas at various pressures.

In an example given, a crude oil residuum was dissolved in propane and the resulting asphaltic residue drawn off. The remaining solution was saturated with gas to a pressure sufficient to cause the separation of a dark constituent. This was drawn off and further saturation carried on, resulting in a series of oil fractionates.

The author compares the effect of cold fractionation with fractional distillation. In the case cited, the fractionates had lower specific gravities and viscosity indexes over 50 units higher than the distillates. An asphalt from a commercial pipe still when treated by this method yielded 14% of a valuable oil.

Natural gas under pressure applied to the cresol-propane extraction system increased the selectivity of the process. When oil dissolved in propane was fractionated by CH_4 in the presence of cresol, the first separation was the cresol phase containing dark-coloured substances.

As the CH_4 pressure was increased to above 40 atm., fractional separation occurred, and under some conditions three immiscible layers were formed. By adding water to the 3-phase system four immiscible layers have been formed, and it is said to be

possible to obtain five immiscible layers in equilibrium by using a solution of ammonia saturated K_2CO_3 .
D. L. S.

164. Oxidation of Naphthene-Base Oils by Oxygen. K. I. Iwanow. *Petrol. Z.*, 1938, 34 (32), 1.—The relative oxidizability of paraffin and naphthene base oils, respectively, was studied in connection with the production of commercial acids from petroleum, and with special reference to the effect of degree of refining prior to oxidation.

Gas oils of the two types were oxidized in a steel bomb heated to 150° C. for 3 hrs. and containing O_2 at 15 atm. Technique of separating unsaponifiable material, fatty acids and oxy-acids is described in detail. Saponification and acid values and iodine value were determined on each type of oxidation product. These methods were applied: (a) to crude distillate and (b) to oils after refining with progressively increasing amounts of sulphuric acid, and finally oleum. A standardized method of refining and washing was adhered to.

It was found that:—

(1) In both types of oil a minimum oxidizability was obtained when about 3% acid was used for refining. This behaviour was attributed to the difficulty of washing out oxidized products after the first acid treatment, these products remaining in the oil as negative oxidation catalysts. Larger amounts of acid destroyed these compounds. With more than 3% acid refining, yield of oxidized products was proportional to degree of refining in both types of oil.

(2) Washing with soda (after acid treatment as opposed to water washing) had practically no effect on oxidizability.

(3) Carboxylic acids formed the chief product of reaction, especially in the more drastically refined samples.

(4) When oxidizability and yield of fatty acids were about the same, less oxy-acids were formed in paraffin base oil than in naphthenic oil. P. G. H.

165. Synthesis of Acetylene. F. Fischer and H. Pichler. *BrennstChemie*, 15.10.38, 19 (20), 377-380.—If an electric arc is burning between two carbon electrodes in a hydrogen atmosphere within a vessel not specially cooled, some percentage of hydrogen is transformed into acetylene and hydrocarbons of high hydrogen content. By cooling the reaction vessel with liquid air, the acetylene formed is frozen out and secondary reactions are inhibited. By this means all hydrogen can be transformed into acetylene and, to a certain extent, into hydrocarbons rich in hydrogen. The reaction velocity depends on the type of electric arc. It is practically independent of pressure, and slows down only at low pressures. In a test, 99.9% of the hydrogen was transformed in 9 min., with a corresponding fall in pressure from 8.8 to 0.007 mm. Hg. There is less tendency to form secondary products of high hydrogen content at low than at high pressures. This is explained as follows: (a) hydrogenation reactions are independent of pressure, and (b) with falling pressure, the probability that a newly formed acetylene molecule, on its way to the wall, may collide with a reactive hydrogen molecule is decreased. Moreover, at the temperature of liquid air, acetylene has the lowest vapour pressure of all hydrocarbons likely to be formed. When using hydrogen-free methane under similar conditions of test, pressure fell within 18 min. from 8 to 0.1 mm. Hg.

Constant-pressure tests were carried out supplying hydrogen through a quartz tube. By this means larger quantities of acetylene could be precipitated on the walls of the reaction vessel. Separation of acetylene from the other hydrocarbons formed is best carried out by pumping out the latter at low temperatures. L. R.

166. Accurate Fractionation of Hydrocarbon Blends. H. Macura and H. Grosse-Oetringhaus. *BrennstChemie*, 1.12.38, 19 (23), 437-439.—The apparatus used consists of a round-bottomed flask of 500 c.c. capacity connected by ground joint to a vertical cylindrical glass tube of 700 mm. height and 24 mm. diameter, with seventeen restrictions to 18-20 mm. diameter for interruption of the reflux current. The lower portion of the column carries a funnel with two openings, and in the top a dephlegmator and a thermometer are inserted. The vapours pass through a vertical cooling coil which condenses the distillate, after which it is collected in graduated receivers. The column is surrounded by a vacuum jacket to prevent heat losses. It is filled with spirals made of 0.5 mm. aluminium wire.

200 c.c. of oil are used for each test or, if contents below 5% have to be determined, 400 c.c. To hydrocarbon blends distilling up to 140° C., 200 c.c. decaline are added, whereas those distilling up to 160° C. are blended with tetraline. Still higher boiling blends are mixed with anthracene or paraffin oil. By this means distillation can be carried out to 95–98%.

Graphs are obtained by plotting temperatures against percentage distilled at 1% intervals. The contents of a certain fraction are found by determining the difference in percentage between the respective points of inflection.

L. R.

167. Applications of Dühring's Rule. E. J. Roehl. *Industr. Engng Chem.*, 1938, **30** (11), 1320.—The Dühring lines for a set of solutions of a given substance form a family of straight lines, each one of which may be characterized by an intercept and a slope. It is shown that logarithmic equations may be used to connect concentration and slope, or concentration and intercept in the case of solutions of sodium chlorate and sodium bromide. If this method of connecting concentration and slope or intercept is generally valid, it is possible to construct a set of Dühring lines for a complete range of concentrations from two measurements on each of two solutions.

Similar equations may be used to connect number of carbon atoms in a homologous series with intercept and slope. This is applied to the paraffin series from propane to nonadecane.

P. D.

168. Separation Processes : Analysis of Unit Sections. M. Randall and B. Longtin. *Industr. Engng Chem.*, 1938, **30** (10), 1188.—The basis of the graphical method of representing separation processes has been previously discussed (*Ind. Eng. Chem.*, 1938, **30**, 1063). The method is applied to various parts of distillation and solvent extraction equipment. As applied to a perfect plate it becomes the well-known method of Ponchon. The authors show how the Murphree efficiency and effect of entrainment may be allowed for. Solvent extraction processes may be similarly dealt with by regarding addition of solvent to be equivalent to addition of heat. Other typical equilibrium processes, such as condensers and vaporizers, are also considered.

P. D.

169. Separation Processes. M. Randall and B. Longtin. *Industr. Engng Chem.*, 1938, **30** (11), 1311.—Previous papers by the authors have discussed the application of molal property *v.* mole fraction diagrams in representing such processes as distillation or solvent extraction. They are not, however, always as convenient to work with as the *x v. y* diagram, and the present paper gives rules and examples of the transformation of one type into the other.

P. D.

170. Phase Equilibria in Hydrocarbon Systems. B. H. Sage and W. N. Lacey. *Industr. Engng Chem.*, 1938, **30** (11), 1296.—The equilibrium constants for methane have been reported for several hydrocarbon systems. In this paper the published data are reviewed and correlated as a function of pressure, temperature, and molecular weight and viscosity-gravity constant of the other constituents.

P. D.

Analysis and Testing.

171. Review of Test Methods for Insulating Oils—The Italian Method. D. Rossetti. *Olii Min.*, 1938, **18** (10), 137–143.—Among the main characteristics which a transformer oil or an electric switch oil has to meet, the tendency of the oil to alteration by effect of temperature and oxidizing action of the air is the most interesting. It is therefore very important to fix by severe and strict specifications a method which permits with sufficient accuracy the reproduction in the laboratory of actual conditions of use.

This method, set forth in its main details in 1924 by the "Regulations for Supply and Control for Transformer Oils and Electric Equipment Oils," was subsequently elaborated to the present method (300-hrs. life test at 110° C. in presence of copper + "Italian Regulations for the Control of Mineral Oils and By-products," second edition, 1928). The results of numerous tests carried out on the same oil in different thermostats of the same type (Heræus) are reported, together with results obtained on identical samples in the same thermostat. An attempt is made to explain the differences found, first with the construction of the thermostats, which, though of the same supplier, are not identical; and then by the fact that the inside temperature is not

equal at all points, because of air draughts circulating in the thermostat. The results of extensive test work demonstrated that while an oil sample placed near the back wall of the thermostat showed after the 300-hrs. life test about 0.45% of sludge deposit and about 0.22% acidity, a sample of the same oil placed near the door of the apparatus showed only 0.35% of sludge deposit and 0.18% acidity. It is concluded, therefore, that given the higher temperature near the back wall, the formation of deposits and acidity is influenced to a greater extent by temperature than by the oxidizing action of the air filtering through the door fissures. In consideration of these factors which affect the results of the life test, it is proposed to use a thermostat of more careful construction, and to adopt a rotating support for eight test-tubes, doing about one revolution per minute, which should be placed inside the thermostat. It is stated that tests carried out with the above-mentioned procedure gave more constant results, showing sensibly lower disparities. Finally, some modifications and closer specifications of the procedure for the official 300-hrs. method are suggested by the author.

P. G.

172. Determining the Sediment Content of Fuel Oil. S. H. Hulse and H. L. Thwaites. *Industr. Engng Chem. (Anal.)*, 1938, **10** (12), 678-680.—No test at present in general use can be employed satisfactorily to indicate the tendency of fuel oils to deposit sludge on storage, or to predict the fuel's stability in contact with preheater surfaces, especially in the case of fuels containing cracked residues. The "sediment by extraction" test is often applied, and does give some indication of storage properties, but is valueless as a guide to stability to heat. The authors' method involves filtering undiluted oil through an asbestos mat in a special steam-jacketed filter funnel, washing the residue free of oil with a high fl. pt. paraffinic naphtha ("Stoddart solvent"), drying and weighing.

The sediment in cracked residues consists partly of small primary particles held together by an asphaltic binder, and partly of solid asphaltic matter, insoluble in oil. The benzene used in the extraction test will dissolve this asphaltic matter and disperse the primary particles, thus giving a totally fictitious idea of the sediment content of the fuel. The new method simulates service conditions which involve heating the fuel, but not thinning it with an aromatic solvent.

The authors quote figures to show that the test is of good reproducibility, the average deviation of eleven laboratories being ± 0.025 in a sediment number of about 0.2, most of which is probably due to the difficulty in getting a truly representative sample.

T. T. D.

173. Iodine Value of Hydrocarbons. H. Grosse-Oetringhaus. *BrennstChemie*, 15.11.38, **19** (22), 417-427.—Based on numerous tests carried out on a variety of oils and gasolines, the merits of the various methods of determining the iodine value are investigated.

In the case of gasolines, the method given by Wijs can only rarely be applied, owing to the great tendency to substitution. The method according to Hanus gives better results. Both methods fail in the case of mineral lubricating oils, but are suitable for highly compounded oils. The iodine values, according to Margosches, are in most cases too low. The lack of a suitable solvent makes application to lubricating oils impossible, except when they are highly compounded.

Kaufmann's method of determining the iodine value of gasolines is considered to be preferable to all other methods, especially in the case of gasolines of high iodine number, if carbon tetrachloride is used as a solvent (reaction time 7-8 hrs.). With lubricating oils only approximate values are obtained.

The method suggested by McIlhiney cannot be applied to mineral oils, no matter what halogen is used. Rhodan (thiocyanogen) values and iodine-rhodan values of gasolines are slightly below iodine values according to Kaufmann. With lubricating oils greater deviations are obtained. The partial iodine value is always considerably lower, and there is no correlation with the thiocyanogen value.

A new method of test is presented—the so-called "iodochloride number"—which is obtained by making use of iodochloride dissolved in methyl alcohol saturated with calcium chloride.

Sources of error and practical applications of the iodine number are given. L. R.

174. Viscosity of Petroleum Emulsions. L. T. Monson. *Industr. Engng Chem.*, 1938, **30** (11), 1287.—Viscosity ratios were measured for twenty water-in-oil emulsions at temperatures of 100°, 130° and 185° F. Five crude oils were used, and emulsions containing 10, 20, 30 and 40% of water were made from each crude. P. D.

175. New Catalyst for the Determination of Nitrogen by the Kjeldahl Method. R. B. Bradstreet. *Industr. Engng Chem. (Anal.)*, 1938, **10** (12), 696.—A mixture of equal parts of ferrous sulphate and selenium is suggested as a catalyst for Kjeldahl nitrogen determinations.

Using sulphuric-salicylic acids as digestion medium, figures are given to show the new catalyst to be at least as good as the copper sulphate-selenium mixture.

T. T. D.

176. Determination of Sulphur in Surface-Active Agents. R. Hart. *Industr. Engng Chem. (Anal.)*, 1938, **10** (12), 688-689.—The methods available for the determination of organically combined SO_3 in detergents or wetting-out agents are: (a) the Herbig method, (b) the acid-titration method, and (c) the ash-gravimetric method, in which the pure sulphonated product is isolated by extracting over concentrated NaCl solution and ashed. The first two methods are of use only in the case of sulphuric acid esters, as true sulphonates are not decomposed under the conditions of the methods. Method (c) overcomes this difficulty, but in the case of highly sulphated oils, the sulphonated alcohols and sulphonated fatty acid amides, the extract carries over with it, in solution, a large amount of salt which vitiates the results.

The new method applies to all compounds that can be quantitatively extracted with a solvent over concentrated NaCl solution. The sample, in a suitable solvent, is shaken repeatedly with NH_4Cl solution until it is quantitatively converted into the ammonium salt—about five operations. The solvent layer is then similarly shaken with concentrated Na_2SO_4 solution; all the ammonia bound to the organically combined sulphate or sulphonate is converted into $(\text{NH}_4)_2\text{SO}_4$, and passes to the water layer, together with any NH_4Cl with which the solvent layer may have been contaminated. The water layer is now analysed for total ammonia, and for ammonia as ammonium chloride. The difference represents the ammonia bound by the organic compound, from which the organically bound SO_3 may be readily calculated.

In cases where they are both applicable, good agreement is obtained between the new method and the ash-gravimetric method.

T. T. D.

Motor Fuels.

177. Anti-Knocking Motor Fuels. M. Marder. *Oel u. Kohle*, 1938, **14**, 697.—A comprehensive survey of available methods for producing fuels of high octane number by solvent extraction, hydrogenation, dehydrogenation, alkylation, cyclization, etc.

The principles underlying each method are described briefly and extensive references are given. The properties and application of the various high octane fuels are outlined.

P. G. H.

178. Patents on Motor Spirit. G. W. Johnson. E.P. 495,889, 22.11.38. Production of motor spirit from gases containing methane homologues which are gaseous at ordinary temperatures. Olefines are first separated, the remainder containing CH_4 homologues is subjected to splitting and the mixture of olefines and split gas polymerized at 350° C. and above 20 atm., in a vessel constructed partly of silicon or of a highly alloyed chromium steel.

Universal Oil Products Co. E.P. 496,273, 25.11.38. Alkylation of *isobutane* and a normally gaseous olefine containing more than C atoms in the molecule, in the presence of aluminium chloride and hydrogen chloride at -50 to 0° C., for the production of high anti-knock motor fuel.

P. Ostergaard. U.S.P. 2,134,836, 1.11.38. Vapour feed condenser and stabilizer for recovering stabilized motor fuel from a vapour mixture containing vapours of normally liquid gasoline components in admixture with vapours of low-boiling hydrocarbons and fixed gases.

D. A. Monro. U.S.P. 2,134,882, 1.11.38. Fractionating apparatus and process for gasolines.

A. L. Lyman and M. M. Holm. U.S.P. 2,135,823, 8.11.38. *iso*-Octane production (octane number above 80), from a normally gaseous hydrocarbon mixture containing olefines consisting substantially of isomeric butenes, by subjecting the mixture to the action of a phosphoric acid catalyst adsorbed on a non-porous, inert solid support at 100–400° F. to effect mixed polymerization of normal butenes and *isobutene*. The polymer liquid is separated from the unpolymerized substances and saturated by dehydrogenation.

E. J. Houdry. U.S.P. 2,136,382, 15.11.38. Catalytic conversion and stabilization of hydrocarbons containing gasoline by contacting, in a desulphurizing zone, with an inert absorbent material impregnated with an active metalliferous material from the group Ni, Co, Cu at 550–750° F. and then passing the hydrocarbon into a refining zone using as catalyst alumina and silica blend and a small quantity of Ni, Co or Cu at 400° F.

P. H. Sullivan. U.S.P. 2,137,825, 22.11.38. Production of motor spirit from normally gaseous hydrocarbons containing CH₄, C₂H₆, C₃H₈, C₄H₁₀, and C₅H₁₂.

P. M. Raigorodsky. U.S.P. 2,138,218, 29.11.38. Method of recovering useful hydrocarbons from gaseous substances—*e.g.*, natural gas, etc.—by an improved rectification process.

W. L. Benedict. U.S.P. 2,138,566, 29.11.38. Removal of copper compounds from copper-sweetened hydrocarbon oils by treating the oil with a double sulphide of Zn and Na in a solid form. W. S. E. C.

See also Abstract No. 179.

Gas, Diesel and Fuel Oils.

179. Proposed Method of Test for Ignition Quality of Diesel Fuels. Anon. *J. Soc. aut. Engrs*, 43, 1938, 453.—The method of test described has been developed by the Automotive Diesel Fuels Division of the Co-operative Fuels Research Committee, and is published for purposes of general information, comments being invited.

The engine specified is the C.F.R. diesel with variable compression. Full details are given of the engine specification and conditions of test. A description of the "coincident flash" apparatus, together with instructions for its operation, are included.

C. H. S.

Lubricants and Lubrication.

180. Recent Physico-Chemical Views on Bearing Lubrication. Burstin. *Petrol. Z.*, 8.6.38, 34 (23), 1–9.—Our knowledge of the mechanical aspect of lubrication has made great progress, but the physico-chemical aspects are only now engaging attention.

The author describes conditions that occur in commercial lubrication ranging from the fluid friction of ordinary bearings to boundary conditions in hypoid gears and the like.

In the latter case it is important that the lubricant should possess good film-forming properties.

The author describes three types of forces which are active in the boundary layer and which arise from the mutual attraction of molecules. These are:—

- (1) The induction effect, which depends on the displacement of the electric charges leading to the formation of an electric moment.
- (2) The dipole effect, caused by the arrangement of the molecules in a definite direction due to the attractive forces of the polar groups.
- (3) The dispersion effect, depending on the effect of electron movement within the atom.

Three types of addition compounds are used to give oils the requisite film-forming properties :—

- (a) Those with carboxyl or ester groups.
- (b) Compounds of Cl₂, S and P.
- (c) Metal compounds, chiefly those of Pb, Cr, Zn and As.

A survey is made of the methods available for testing such oils. Amongst those described are the "ferrum reductum" test of Irauth and Neyman, the S.A.E. and Timken machines, the Thoma and Spindel testers, and the instruments designed by Kyropoulos, Redgrove and Ralston, Hoffman and Stephens.

Finally the "Frikto-meter" of Wilder is described in detail and the method of using it to determine the static friction is discussed.

D. L. S.

181. Viscosity Measurement on Lubricating Oils Below 0° C. K. Schwaiger. *Petrol. Z.*, 1938, **34** (45), 1.—A new type of viscometer to deal with oils at 0° C. to -5° C. is described. An inner cylinder, capable of rotation, is fitted into another cylinder. The bearing surfaces (0.05 mm. clearance) are wetted with the oil under test. The inner cylinder may be rotated by a fixed weight and pulley system. The whole apparatus is cooled in a chamber which forms part of a refrigerator. The time for one half revolution of the inner cylinder is multiplied by a factor which gives dynamic viscosity in centipoises directly.

Viscosities and pole-heights obtained by extrapolation of values determined by normal methods at higher temperatures deviated from those obtained by the new viscometer. This is stated to show that the Walther relationship is inapplicable at low temperatures. Values obtained by the author's "practical" method and by Vogel-Ossag method respectively on five oils of very low setting point differed considerably in the case of three of the oils below 0° C.

The energy consumed by a starter motor in turning an actual engine at low temperatures was measured and the viscosity of lubricating oil in the engine cylinders was calculated. The viscosity curves for several oils obtained by the motor method were closely parallel to those given by the author's viscometer.

P. G. H.

182. Oxidation of Roumanian Lubricating Oils. C. Candea and C. Manughevi. *Petrol. Z.*, 1938, **34** (49), 4.—Roumanian oils were oxidized with air in the presence of 0.02% of various inhibitors (thymol, *p*-toluidine, naphthylamine, α -naphthol, hydroquinone, phloroglucinol and resorcinol). Tests were carried out at 100° C., 200° C. and 300° C.; peroxide no. (Yule and Wilson), acid value and saponification value were determined after oxidation. Peroxide formation, which is regarded as the first stage of oxidation, occurred most readily at 200° C., but in all cases was lessened by the inhibitors. Development of acidity was also retarded by the inhibitors. In most of the tests, saponification value was reduced by the inhibitors at 200° C., but not at 100° or 300° C.

Thymol was the most effective inhibitor at 200° C.; its optimum concentration being 0.4% at 300° C., however, the maximum effect was obtained with 0.2% thymol.

P. G. H.

183. Colloidochemical Basis of Greases. E. H. Kadmer. *Petrol. Z.*, 8.6.38, **34** (23), 2-4 (Suppl.); 13.7.38, **34** (28), 2-4 (Suppl.).—Emulsifying agents like soda soaps which lower the surface tension of water more than oil result in oil-in-water emulsions, whilst soaps such as those of lime and aluminium which lower the surface tension of oil more than water form water-in-oil emulsions. Thus lime base greases are of the water-in-oil type, whilst soda base greases belong to the reverse type. The author reviews briefly the commercial methods of manufacture of Na, Ca, Al and Pb greases.

The stability of lime-base greases can be improved by increasing the viscosity of the oil base and by using a proportion of neutral fat with the fatty acids. In the case of soda greases, the sponge type possesses a structure very difficult to modify by mechanical means. The dropping point of a soda soap lies between 230° and 270° C., and that of the grease prepared from it depends on the quantity of soap it contains, the nature of the fat from which the soap was made, etc.

Commercial aluminium greases, according to the author, cannot be made from an aluminium soap alone. Additions are recommended, and three are mentioned in this paper: (a) wax (Montan, wool, etc.), (b) crepe rubber, (c) rubber latex.

There is an urgent need for a water-resisting grease with a drop point of 120–130° C., and the author suggests that mixed Na and Ca base greases which are sometimes used for this purpose are not always successful. He suggests the use of mixed Na and Al base products, since these are both more or less water free. D. L. S.

184. Patents on Lubricating Oil and Wax. T. N. Montgomery and I.C.I., Ltd. E.P. 495,410, 14.11.38. Stabilization of chlorinated paraffin wax by adding 0.5–1% of a hydrocarbon of the pinene or terpene group or mixtures of these.

Robinson Bindley Processes, Ltd., and W. W. Myddleton. E.P. 495,575, 16.11.38. Catalytic synthesis of hydrocarbon oils from CO and H₂ in which the catalyst is used in porous spaces form and arranged between two concentric cylindrical surfaces. The rate at which the gases are passed over the catalyst is also specially controlled.

Standard Oil Development Co. E.P. 495,869, 18.11.38. Oxidation inhibitor for mineral lubricating oils consisting of 0.4% by weight of di-(4-tertiary butyl-6-hydroxyphenyl)thioether and 0.2% by weight of triphenyl bismuth or a mixture of 0.4% by weight of di-(4-tertiary butyl-6-hydroxyphenyl)thioether and 0.2% by weight of triphenyl tin iodide.

Metallgesellschaft, A.-G. E.P. 495,949, 22.11.38. Distillation apparatus for high-boiling liquids.

Colgate-Palmolive-Peet Co. E.P. 496,175, 25.11.38. Distillation apparatus for high-boiling liquids—*e.g.*, fatty acids.

W. G. Hiatt. U.S.P. 2,134,824, 1.11.38. Desulphurizing of petroleum oils by separating the vapours boiling between 600 and 800° F. and treating them with an inert gas heated to this temperature, and passing the diluted vapours in contact with a catalyst of the clay type.

M. Landau. U.S.P. 2,135,009, 1.11.38. Process of separating hydrocarbon mixtures into fractions using benzene as an extraction agent.

R. de M. Traveau, L. A. Clarke and R. E. Manley. U.S.P. 2,135,363, 1.11.38. Deasphalting of hydrocarbon oils using *isopropyl* alcohol, butyl alcohol and furfural.

G. H. Morley. U.S.P. 2,135,468, 1.11.38. Solvent refining of hydrocarbon oils using nitro-alcohols—*e.g.*, 3-nitro-4-heptanol.

E. Terres, J. Moos and H. Ramser. U.S.P. 2,135,922, 8.11.38. Dewaxing of lubricating oil using sulphuric oxyfluoride and an auxiliary solvent—*e.g.*, benzol.

R. C. Powell. U.S.P. 2,136,172, 8.11.38. Vacuum distillation apparatus for lubricating oils.

H. R. Dickinson. U.S.P. 2,136,282, 8.11.38. Process of imparting amorphous characteristics to normally crystalline waxes.

E. Terres, J. Moos and H. Ramser. U.S.P. 2,136,767, 15.11.38. Dewaxing of lubricating oils using an aromatic fluorine compound and benzol as auxiliary solvent.

F. X. Govers. U.S.P. 2,137,206, 15.11.38. Solvent refining of hydrocarbon oils using methyl furfural.

J. H. Grahame and W. L. Douthett. U.S.P. 2,137,207, 15.11.38. Dewaxing and deasphalting of hydrocarbon oils using propane at 80–100° F.

H. H. Gross and W. Kierstead, Jr. U.S.P. 2,137,208, 15.11.38. Dewaxing of hydrocarbon oils using a mixture of 40% of methyl ethyl ketone and 60% of benzol at 160–170° F.

E. C. Knowles. U.S.P. 2,137,209, 15.11.38. Dewaxing hydrocarbon oil in which the oil and wax are mixed with a selective solvent—*e.g.*, acetone and benzol.

F. X. Govers. U.S.P. 2,137,218, 15.11.38. Dewaxing of lubricating oil using a mixture of SO₂ and methylene chloride.

R. Z. Moravec. U.S.P. 2,137,499, 22.11.38. Solvent extraction of hydrocarbon oil by contacting the oil with a tertiary aliphatic alcohol and methyl ethyl ketone under conditions causing the formation of two liquid phases containing alcohol, ketone and different portions of the oil.

P. Subkow. U.S.P. 2,137,549, 22.11.38. Dewaxing of hydrocarbon oils using trichlorethylene, SO₂, benzol, CCl₄, etc.

A. W. Hixson and R. Miller. U.S.P. 2,138,166, 29.11.38. Solvent refining of hydrocarbon oils using chloronitropropane or chloronitrobutane.

E. Saegbarth. U.S.P. 2,138,772, 29.11.38. Refining of lubricating oils using *p*-monochlorophenol in the presence of propane as diluent.

E. Terres, J. Moos and E. Saegbarth. U.S.P. 2,138,773, 29.11.38. Solvent extraction of lubricating oil using benzol and acetone at 108° F.

C. C. Towne. U.S.P. 2,138,775, 29.11.38. Preparation of synthetic lubricating oil by condensing a mixture of 8 parts by weight of propylene chloride and 10–12 parts by weight of benzene in the presence of 1–2 parts by weight of aluminium chloride at temperatures below 60° C. W. S. E. C.

Asphalt and Bitumen.

185. Compressibility and Ductility of Filled Road Tars. Anon. *Asph. u. Teer*, 1938, **38** (47), 800–806.—This paper describes investigations in which the effect of ageing and the incorporation of various fillers on rate of evaporation, compressibility and ductility of road tars and the relative wetting power of the tar for the fillers was examined. Rate of evaporation was determined at various temperatures and times by estimating loss in weight of films of tar 0.15 mm. thick on glass plates. For the wetting power a drop of the tar was allowed to fall from a Ubbelohde drop-point apparatus on to the filler, which was placed on a glass plate suspended in water at 55° C., the area of the drop of tar then being determined after cooling. Compressibility was determined by measuring the decrease in height at various temperatures, under a weight of 10 gms., of specimens 50 mm. high and 10 mm. in diameter. In the ductility test one end of the usual mould is fastened to the movable carriage and the other to a cord attached to a weighted lever. The two latter tests are considered more suitable for examination of the effect of ageing and filler than the method proposed by Sabrou and Renaudie. A. O.

186. Viscosity and Cohesion of Bituminous Binder in Relation to Temperature. G. Zichner. *Asph. u. Teer*, 1938, **38** (49), 835–841.—It is pointed out that in the investigation of physical-mechanical properties of binder-aggregate mixtures, conditions must be so controlled that only the binders differ. The object of the investigation described was to examine the relation between viscosity and cohesion. The various commercial methods of determining viscosity of bituminous binders were considered unsuitable, and the Couette apparatus was first employed in the investigation, but was unsuitable for determinations at high temperatures, and the Höppler falling sphere viscometer was eventually used. Results for a number of binders indicated that a straight line was given when the viscosities divided by a constant were plotted on the log log viscosity-log absolute temperature system, the constant for road tar being 2.0 and for bitumen 1.75. It is not known whether these constants hold for all materials. It is suggested that all binders be characterized by the temperature at which the binder has a definite viscosity, and the temperature susceptibility by the temperature interval necessary to increase the viscosity from 80 centistokes to 200,000 centistokes. Cohesion measurements were made on binder films and binder-aggregate mixtures by determining the load required to pull apart the films or mixtures compressed between metal plates; the apparatus is described. It is concluded that the fracture of binder films is generally due to other more complicated phenomena than the overcoming of cohesion. A cohesion fracture is obtained only with binders of high viscosity. A. O.

187. Crystals Obtained from Asphalts. R. Ariano. *Strade*, 1938, **16**, 556–561.—The presence of crystals has been observed on the surface of asphalts obtained from Abruzzi asphalt, and it was suggested that this asphalt contains saturated solution

of a substance which, with increase of temperature, distills off and collects on the surface of the asphalt, thus giving rise to formation of the so-called "efflorescence." A method has been devised for collecting sufficient of the substance for analysis; this consists in extraction of thin films of asphalt under vacuum. The substance proved to consist of very small sulphur crystals. Research work carried out also on asphaltic dusts of different origin showed that the formation of crystals took place only when the extracted asphalt contained over 20% of sulphur, which is about the concentration of saturation. The phenomenon does not occur in petroleum bitumens, owing to their lower sulphur content. It has been observed, however, that petroleum asphalts also form crystals when the sulphur content is increased to 20%. The presence of the crystals does not cause inconvenience in actual use. P. G.

188. Affinity of Hydrophilic Aggregate for Asphaltic Bitumen. H. F. Winterkorn. *Industr. Engng Chem.*, 1938, **30**, 1362.—An attempt is made to determine the various factors which are of greatest importance in the adhesion problem as applied to bituminous road construction. The influence of different pretreatments on the resistance to stripping of bitumen-coated aggregate was determined by means of a wash test. The various pretreatments included washing of the aggregate (before coating with bitumen) with distilled water, aqueous hydrochloric acid, and aqua regia, respectively, and then priming with aniline, phenol, furfural, aniline-furfural, phenol-furfural and lead acetate, respectively. In some cases the wash test was preceded by curing periods in air or in water. The data obtained indicated that:—

1. The stripping-resistance of bitumen-coated aggregate depends on the properties of the bitumen, the surface properties of the aggregate, and the time and condition of contact between mineral surface and bitumen.

2. The effect of acid washing before coating depends on the character of the mineral surface and on the bitumen, but aqua regia is superior to hydrochloric acid as regards improving the adhesion.

3. Priming with furfural and its derivatives is beneficial in improving resistance to stripping.

The evidence accumulated tends to show that furfural and its resinous derivatives might be destined to fill an important place in bituminous road construction.

H. E. T.

189. Asphalt and Bitumen Paints. F. Ohl. *Asph. u. Teer*, 1938, **38** (45), 761-765.—Natural asphalts, such as Gilsonite, are preferred for manufacture of high-quality paints and enamels, but other materials employed, either alone or blended, include coal-tar pitch, oxidized coal-tar pitch, petroleum bitumens, blown and high-vacuum bitumens, stearine and wool pitches and mixtures of bitumen and chlorinated rubber. High-vacuum bitumens are said to be more stable to light than other so-called artificial bitumens, whilst blown bitumens become brown and chalky in direct sunlight, but are superior to the former where the film is subject to bending or shocks. Owing to the large amounts of mineral colour which are necessary in coloured paints, they lack stability; aluminium powder is the most satisfactory and has good protective properties. Bitumen paints should not be used on zinc, owing to the formation of acidic weathering compounds. Bitumen enamels may contain only bitumen and a volatile solvent, or may contain bitumen, resins, softeners, etc., with or without a volatile solvent. Turpentine is not used in linseed oil-bitumen enamels. Essential properties of bitumens for use in paints and enamels are hardness, black gloss, resistance to chemical action, easily and completely soluble, ash content less than 0.2% and wax content less than 2%. Larger amounts of wax cause the surface to become dull and wrinkle. In suitable bitumens the difference between softening point and drop point is about 20° C., and the length of the drop-point thread about 18 mm. Typical formulæ for paints and enamels are given. A. O.

190. Patent on Asphalt. H. E. Potts. E.P. 495,229, 9.11.38. Production of bituminous emulsions free from fillers or solid dispersing agents, having a high stability towards lime and containing 0.6-1% of alkaline metal salts of oxalic acid.

W. S. E. C.

Special Products.

191. Ichthyol—Its Source and Properties. O. C. Blade. U.S. Bur. Mines. Inf. Circ. 7042.—The term Ichthyol is a name applied loosely to certain pharmaceutical preparations manufactured by various chemical processes. The correctly applied word refers to the product obtained by sulphonating and then neutralizing with ammonia a distillate from the oil retorted from a certain high-sulphur bituminous shale found near Seefeld in the Austrian Tyrol. An account is given of the physical and chemical properties of bituminous shales in the Tyrol and of the oils obtained from them. The deposits are exploited by modern mining methods, but little actual data are known concerning the processes of retorting the shale. Ichthyol is a soft, semi-solid, dark-brown material with a characteristic bituminous odour and a burning taste. It has a specific gravity of approximately 1, is soluble in water and partly so in alcohol or ether alone and completely in a mixture of equal volumes of ether and alcohol. It is largely used as an external application for skin diseases and inflammations. Numerous modifications of Ichthyol have been developed for specific purposes—*e.g.*, Ichthargan—obtained by treating the sulphonated product with silver oxide; Ichthalbin, the product of the sulphonated material and albumen, and Ichthoform, the formaldehyde reaction product of the acidified oil. B. M. H. T.

192. Diisobutylphenol. J. B. Niederl. *Industr. Engng Chem.*, 1938, **30** (11), 1269.—The physical, chemical and physiological properties of diisobutylphenol (*p*-*tert*-octylphenol) are described, and conclusive proof of its structure is given. Diisobutylphenol is now manufactured in America on the industrial scale. Many new derivatives have been prepared, including new dyestuffs, a new salicylic and aspirin analogue, new long-chain substituted alicyclic compounds, as well as a similarly substituted aliphatic dibasic acid, and also numerous aromatic compounds, all possessing the diisobutyl radicle in the side-chain. The diisobutylphenol and consequently all its derivatives are prepared from phenol and diisobutylene. H. E. T.

193. Patents on Special Products. Chemische Werke vorm H. & E. Albert. E.P. 495,192, 8.11.38. Manufacture of alkaline polyphosphates for use as detergents, washing and softening agents.

Société Carbochimique, P. Ferrero, C. Vaneridries and F. Berbe. E.P. 495,676, 17.11.38. Preparation of ethylene oxide and its homologues from aqueous solutions of the corresponding chlorohydrins by hydrolysis with an alkaline or alkaline earth base.

F. W. Corkery. U.S.P. 2,135,423, 1.11.38 and U.S.P. 2,135,430, 1.11.38. Production of residual elastic gums from liquid still residue from coke-oven light oils by oxidation, without substantial polymerization.

F. W. Corkery. U.S.P. 2,135,427, 1.11.38. Purifying raw aromatic still residue from the treatment of coke-oven light oils by solvent separation and sulphonate precipitation.

F. W. Corkery. U.S.P. 2,135,429, 1.11.38. Purification of still residue for use in resin production. W. S. E. C.

Detonation and Engines.

194. Use of 59 O.N. Motor Spirit in High Compression Engine Developed for Use with Wood Producer. M. Puisais. *Ann. Off. Combust. liq.*, 1938, **13**, 299–312.—The author calculates that the use of producer gas derived from charcoal, in an engine of 8 to 1 compression, will give 34% less power than the same engine when operated on motor spirit, the compression ratio in the latter case being 6 to 1.

Actual experiments indicated that a loss of 42% was obtained, which is ascribed to the differences in the combustion speeds in the two cases, the combustion with motor spirit being the faster. In order to obtain the same power using the charcoal producer, it is necessary to increase the capacity of the engine 1.6 times.

The problem of running such an engine of 8 to 1 compression with 59 O.N. gasoline can be solved by fitting the engine with a regulator which limits the pressure of the

air-fuel mixture before compression to about 44 cm. Hg. If it is desired to use present engines to run on producer gas, supercharging will be necessary in order to obtain the same power output as that available with motor spirit. W. E. J. B.

195. The Maintenance of Diesel Plant at the Stations of the British Broadcasting Corporation. L. Hotine and P. H. Smith. *Diesel Eng. Us. Ass.*, Report No. S. 146.—A description is given of the running and maintenance of twenty-five diesel engines installed at the various stations of the B.B.C.

Some special methods adopted to reduce cylinder wear and piston-ring sticking and to control oil consumption are discussed, and information is given regarding the effectiveness of these measures.

A theory to account for cylinder wear is developed in which the cylinder liner surface is assumed to form itself into "striations" on account of temperature stresses. Photographic evidence in support of this theory is given. The design of the upper extremity of the liner is stated to determine whether or not "striations" will be serious, and some successful modifications are described, based on the principles of the authors' theory, which have, in practice, reduced "striations" to harmless proportions. J. G. W.

196. Diesel Engines and the World's Fuel Supply. J. J. Broeze and J. O. Hinze. *Diesel Eng. Us. Ass.*, Report No. S. 147.—The relation between the diesel engine and its fuel is examined, and it is shown that much would be gained if a more universal attitude were adopted towards designing engines to consume existing cheap fuels, rather than to adapt the fuel to suit individual engines.

Fuel qualities which control the digestibility in an engine are discussed, together with the reliability of certain laboratory tests in assessing the performance in the engine. Engine characteristics, such as the degree of cooling given to the spray nozzle, are shown to affect the combustion of the fuel to a marked degree, bad combustion also leading to other indirect complications. The advantages to be gained by cooling the spray nozzle in order to avoid carbonization are stated to be very great, and a design in which the nozzle is cooled by the fuel is illustrated.

Some general details of a design of engine are put forward by the authors, which, in their opinion, would be practically omnivorous in regard to its fuel. J. G. W.

197. Penetration of Oil Sprays. P. H. Schweitzer. *J. Appl. Phys.*, 1938, 9 (12), 735.—A method of measuring the penetration of a fuel spray tip is described in which the spray tip is made to impinge on a sensitive electric contact. By this means the effect of injection pressure and orifice diameter on spray tip penetration were measured and the results obtained used to check the theoretical dimensional relationships.

Investigations on spray velocity were conducted using a ballistic pendulum, with a specially developed shield to avoid the impact effect of the air carried along with the spray.

Conclusions are reached from the data obtained which lead to certain general relationships between the issue velocity, injection pressure, hole size, oil viscosity, chamber air density, air resistance, and spray penetration. J. G. W.

198. Automotive Two-Cycle Diesel Engines. F. G. Shoemaker. *J. Soc. aut. Engrs*, 1938, 43, 485-495.—The problems involved in designing an automotive diesel engine that compares favourably with conventional gasoline engines as to size, weight and power lead the author to the belief that the two-cycle engine is the solution, and the paper deals with the design of such a type of engine using similar materials, manufacturing methods and parts as are common practice in gasoline engines.

The engines described are those of the General Motors, Series 71, uniflow two-stroke range, which are built in three sizes—viz., 3-, 4- and 6-cylinder models of 82.5, 110 and 165 maximum B.H.P., at 2000 r.p.m., respectively. A three-lobe helical blower is used for pressure scavenging through piston-controlled intake ports and overhead poppet exhaust valves.

Piston cooling is obtained by an oil spray, and by this means and by coring out the rim of the piston between the piston crown and the ring belt it is claimed that ring-belt temperatures are obtained well below the coking or gumming point of ordinary oils. The injectors are of the unit type operated by rocker arm and push rod from the cam

shaft, and injection pressures vary from 10,000 to 25,000 lb. per sq. in., the plunger being $\frac{3}{8}$ in. in diameter with a displacement of 501 mm.³. The symmetry of design of these engines allows the cylinder block and/or cylinder head to be turned end for end, thus permitting the accessories and exhaust manifold to be fitted on either side, regardless of the direction of engine rotation, which can be reversed by fitting the idler gear on the other side of the balanced shaft and cam shaft which are interchangeable to permit reversing the cylinder head.

A comparison between gasoline, four-cycle diesel and two-cycle diesel engines shows that in the case of the six-cylinder engine the two-cycle design is equal in weight per B.H.P. to the gasoline, and lighter and smaller than the four-cycle diesel engine.

C. H. S.

199. Some Factors Controlling Part-Load Economy. H. Rabezzana. *J. Soc. aut. Engrs*, 1938, **43**, 511-514 and 528.—The author contends that any further worth-while improvement in economy of modern automobile engines is likely to be realized only if improvement in relatively small details can be effected.

The main disturbing factors inherent in the majority of modern engines are :—

- (1) Non-uniform mixture distribution.
- (2) Low mixture speed in manifold on light loads.
- (3) Poor turbulence in cylinder head.
- (4) Stratification of the mixture.
- (5) Dilution of mixture due to valve overlap and exhaust back pressure.
- (6) Poor scavenging around spark plug.
- (7) Improper mixture at the spark-plug gaps.

Each of these points is dealt with, details of tests illustrating them being given, and methods of improving the shortcomings suggested.

C. H. S.

200. Spark Advance and Octane Number—A Road Test Technique. W. E. Drinkard and J. B. Macauley, Jr. *J. Soc. aut. Engrs*, 1938, **43**, 436-440.—This article describes a method of investigating fuel-car relationship by an improved knock die-out determination technique.

By using a spark advance mechanism operated from the driver's seat and a suitable spark advance indicator, it is possible to obtain curves showing the speed at which knock disappears at any spark setting. Such data are presented in respect to blends of reference fuels and several commercial gasolines.

Knowing the spark advance characteristics of the distributor when operating automatically, and the tolerances allowed by the makers, it is possible from such data to predict the performance of a car as regards knock with its normal ignition equipment.

The data include the effects produced by operating on various fuels with the distributors working automatically but with different basic settings.

It is claimed that by the methods described variations between cars of the same model caused by different distributor characteristics, varying carbon deposit and scale, etc., can be fully investigated.

C. H. S.

201. Correlation of Road and Laboratory Octane Numbers. J. R. Sabina. *J. Soc. aut. Engrs*, 1938, **43**, 416-420.—This report submits the findings of the Correlation Group of the Co-operative Fuel Research Committee, which, at the November 1936 meeting, was authorized to make a study of the correlation between laboratory and road knock-ratings in the then current automobiles. A preliminary study based on information submitted by individual Companies in 1934, 1935 and 1936 cars indicated the desirability of collecting further data under controlled conditions. The results of that work have already been presented in September and October 1937. The Correlation Group have also analysed the report of the Institute of Petroleum Knock-Rating Panel based on British cars and fuels, and have concluded that the same factors influence both the British and American data in about the same degree, and assume, therefore, that any laboratory method of test which is shown to improve the correlation between road and laboratory ratings regarding American cars will apply in like degree to the British cars.

The best correlation appears to be obtained by taking a mean of the research and motor method ratings as the laboratory rating, but in view of the complication of using

two methods, the question of relative reproducibility and the extent to which the fuels tested are representative of commercial gasolines, the Group recommend that no change from the motor method be made at present. C. H. S.

202. Application of Statistical Concepts to the Knock-Rating Problem. J. M. Campbell and W. G. Lovell. *J. Soc. aut. Engrs*, 1938, **43**, 421-426.—From the available data on road and laboratory knock-ratings, the authors have begun a statistical analysis to determine the probabilities of occurrence of errors of various magnitudes. They find the random error among successive measurements in road tests to be from three to six times that between laboratory ratings in the C.F.R. engine, and the variability among ratings made in different makes of car only slightly greater than that among repeated tests in a single car of one make.

This high random error in car ratings makes it necessary to obtain a large number of road ratings on any particular gasoline before a reliable comparison can be made between it and another fuel with the same precision that is obtained in the mean of three or four laboratory ratings on each fuel. C. H. S.

203. Practical Approach to the Road Detonation Problem. A. J. Blackwood, C. B. Kass and G. H. B. Davis. *J. Soc. aut. Engrs*, 1938, **43**, 427-433.—The authors contend that the average fuel octane-number requirement of existing cars and the octane requirement of the average car are values of little practical significance. They show how the road octane number of a fuel varies in some cases when tested at varying knock intensity, and how the spread of ratings of a fuel in a series of cars gives no indication of comparative behaviour, as a value of, say, 70 octane number may have been accompanied by objectionable knock, whereas a rating of 65 octane number in another car may have represented only a trace knock.

For these and other reasons described it appeared desirable to tackle the problem of evaluating fuels from another angle. The procedure adopted was to select a large number of privately owned cars, and tests were made on reference fuel blends and branded products, with the idea that if the cars selected constituted a representative cross-section of the total cars on the road, then the results obtained should be representative of actual service conditions.

Two such practical surveys were made, and a statistical analysis of the results is given. The data were considered on the basis of their representing 100% of the cars on the road, each of the 265 cars tested being given a value (calculated from the number produced, mortality rate, etc.) representing its proportion of all cars on the road. Cumulative curves are given showing the percentage of cars which will give varying intensities of knock on a reference fuel blend of a given octane number, and the knocking tendencies of six non-premium fuels are shown in a similar way. These six fuels differ markedly in the percentage of cars in which they develop a given knock intensity. These differences constitute a basis for the development of a practical method of relating laboratory and road ratings, since the best laboratory method should be one which shows a corresponding linear increase in the percentage of cars satisfied as the numerical value of the laboratory rating increases. The results on these six fuels are shown when the laboratory ratings by three different methods are used. It is concluded that laboratory ratings are useless unless they predict relative performance of fuels in the cars actually being operated by the public. As a result of these two surveys it is felt that this method of test yields data of practical value.

C. H. S.

204. Rating Aviation Fuels in Full-Scale Aircraft Engines. H. K. Cummings. *J. Soc. aut. Engrs*, 1938, **43**, 497-503.—The first report of the Co-operative Fuel Research Committee dealt with the results obtained on three series of base fuels in four representative full-scale aircraft engines and assignments were made for further work as follows:—

- (1) Establish the validity of the C.F.R. Recommended Procedure for rating fuels above 87 octane number in full-scale engines.
- (2) Conduct full-scale engine tests in the range from 87 octane number to the highest octane number available.
- (3) Concurrently with Item 1, develop or revise knock test methods leading to correlation with full-scale engine data.

The present report covers work under Items 1 and 2 and presents partial data required under Item 3.

Since presentation of the first report the National Research Council of Canada have become a co-operating member of the group, and have carried out tests which are described, using a 14-cylinder Armstrong-Siddeley "Jaguar" engine. The results of these tests are in substantial agreement with the work in the first report. The Wright supplementary tests of olefinic fuels show that the A.S.T.M. method satisfactorily predicts the full-scale value of very highly unsaturated fuels of both leaded and unleaded type up to 84 A.S.T.M. octane number.

The tests of commercial 87 A.S.T.M. and 100 Army octane-number fuels containing $2\frac{1}{2}$ -3 c.c. of PbEt₄ show that the A.S.T.M. method underrates 87 octane leaded fuels by 6 to 8 octane numbers, and 100 octane leaded fuels by 2-4 numbers. From the work of both reports the following tentative conclusions are arrived at.

Unleaded *iso*-octane blends of 84 A.S.T.M. octane number or more will be lower in full-scale rating than leaded blends of equal A.S.T.M. rating containing over 2 c.c. of lead per gallon.

Benzol blends generally will not be equal in full-scale rating to leaded blends of equal A.S.T.M. rating if the A.S.T.M. rating is over 84. When benzol blends of 84 or more octane number are compared with *iso*-octane blends of equal A.S.T.M. rating, they will on the average be higher, and rarely lower, in full-scale rating. The behaviour of high-octane-number benzol blends in full-scale engines is widely variable as a result of engine type and operating conditions. It is apparent, however, that any revised knock-test method should not be more severe on aromatic blends than the A.S.T.M. method.

Regarding assignments 1, 2 and 3, it is found that :—

(1) The recommended procedure of bracketing the test fuel between blends of special reference fuels M.1 and S.1 is satisfactory.

(2) Before 100 octane-number fuels employing bases of diverse chemical type can be used in service with reasonable safety extensive full-scale investigations are necessary.

(3) Revision of the A.S.T.M. method is necessary. As a result of extensive studies, a Laboratory Procedure Group is considering immediate modification, which it is believed will give results more in line with full-scale practice than the A.S.T.M. method.

C. H. S.

Coal and Shale.

205. Modern By-Product Coking Design. Anon. *Fuel Econ.*, 1938, **156** (14), 327.—The article describes the evolution of the modern coke-oven, laying particular stress on the early work of Koppers.

Advances in oven design in practice have been chiefly in the direction of a reduction of operating costs and an improved thermal efficiency. These points are shown in the design of a modern combination circulation oven, which is described and illustrated in the article.

W. E. J. B.

Economics.

206. World's Petroleum Industry in 1938. Anon. *Przem. Naft.*, 10.1.38, (1), 13-15.—The paper gives a review of political happenings in the past year and discusses their influence on the oil industry and trade. It is stated that the general lowering of the production in 1938 is by no means an indication of an exhaustion of the existing oil resources.

Tables are given showing the prices of petroleum products (in U.S.A. only) and the approximate production in 1938 of most of the oil-producing countries. E. J. W.

207. Diesel Propulsion in the Development of Communications. Anon. *Przem. Naft.*, 10.12.38 (23), 635.—The increase in importance of the diesel engine over the spark-ignition engine is mainly due to three factors: (a) the high thermal yield of the diesel engine; (b) the low cost of the fuel; (c) the very large range of mechanical power for which diesel engines can be used. The maximum power developed by spark-ignition engines is 3000 Mech. H.P., as compared with 25,000 Mech. H.P. developed by diesel engines used in the shipping and rail industry.

Shipping. In the last twenty-five years the ratio of the tonnage of diesel-propelled ships to the total tonnage of the world's mercantile fleet rose from 0.45 to 22.5% and a further increasing tendency is observable.

By the end of September 1938 out of 685 ships being built all over the world 433—i.e., 65%—were equipped with diesel propulsion.

Of the shipbuilding countries, Denmark heads the list with 100%, which means that all the ships built there are motor propelled. Italy, Sweden and Holland follow next. Great Britain is in the sixth place with 57.2%.

Rail Traffic.—Rail cars and diesel-propelled locomotives are gaining in popularity, particularly for local traffic. In a given table Germany heads the list with 671 rail cars and 1171 diesel locomotives, followed by France, Argentine and Italy.

Road Traffic.—Here the competition between the railways and mechanized road transport has an inhibiting effect. The price ratio of gas oil to petrol seems to have a determining effect on the development of diesel lorry transport.

Air Traffic.—Here also the above ratio plays an important rôle. The considerable weight of the diesel engine is a disadvantage. Since 1929 experiments were being carried out in Germany with diesel aero-engines, and in 1932 the first passenger plane with diesel propulsion was put into regular service. Satisfactory results in this sphere were also obtained in the U.S.A. in 1931.

In the course of the last five years it was possible to raise the efficiency of diesel aero-engines from 25 to 40 Mech. H.P. per litre, lowering at the same time their weight from 1.1 to 0.6 kg. per Mech. H.P. E. J. W.

208. Estimated Cost of a Five-Year Drilling and Prospecting Plan in the Carpathian Area. M. Mrazek. *Kop. Naft. Polsce*, November 1938, **11**, 332.—In connection with a five-year plan for drilling and prospecting work in the Polish Carpathian area the author gives an estimate of the costs. Tables are given showing the dimensions, quantities and cost of drilling equipment, casing and tubing as distributed over five years. Other tables give in detail the cost of different factors involved in drilling and prospecting. The total cost as estimated amounts to 75,087,000 zlotys. E. J. W.

209. Five-Year Plan for Prospecting and Drilling Work in the Carpathian Area. K. Tolwinski. *Kop. Naft. Polsce*, November 1938, **11**, 334-336.—Poland's oil production is steadily decreasing, although the number of bore-holes increases. This leads to the conclusion that the resources of the present oil-fields are being exhausted. Therefore the necessity arises to develop intense drilling and prospecting activities in the Carpathian Area, where there is strong evidence that new oil-fields can be found. The author suggests a programme of work extending over five years. The area is divided up into five regions, in which drilling and prospecting equipment is distributed according to the requirements of the geological structure and complications that are likely to be encountered. E. J. W.

210. Reorganization of Mechanical Transport in Czechoslovakia. Anon. *Przem. Naft.*, 25.12.38 (24), 660.—The far-reaching changes in the geographical and economic structure of Czechoslovakia have brought about the necessity of a reform of the mechanical transport of the country.

The main decisions that have been taken by the government in this connection are these :

- (1) Abolition of all taxes on motor-cars of any type from January 1st, 1939.
- (2) Abolition of all restrictions in the sphere of so-called Industrial Road Transport (car park taxes, etc.).
- (3) Abolition of a number of limitations of the commercial goods transport. No restrictions are made for the freight charges of lorries below 3 tons capacity. Lorries of over 3 tons capacity, however, are brought under the direct or indirect control of the railways. As a result of this restriction the industrial goods transport of individual companies is almost entirely limited to rail transport.
- (4) In bus passenger transport the tax on each ticket is reduced from 20 to 10%.

Parallel with these reforms, the existing road system is to be enlarged. E. J. W.

History of Petroleum.

211. **History of Petroleum in Persia to the Beginning of the Twentieth Century.** L. Lockhart. *Rev. Pétrolif.*, 4.11.38 (810), 1421-1425; 11.11.38 (811), 1455-1459.—Archaeological research shows that bitumen was used in the Sumerian epoch (5000-6000 years ago). It was used as a binder, in making ornaments, for waterproofing pottery and for caulking. The investigation of the use of liquid petroleum is more difficult, and depends on documental and philological evidence. The word naphtha is of oriental origin, but whether Persian or Semitic is debatable. It certainly meant petroleum in ancient Persia. Gas issues were important, being employed in religious rites. However, geological evidence is against the existence of a seep on the site of a temple at Masdjid-i-Sulaiman (596 B.C.). Crude oil is unlikely to have been burned in the temples because of the fumes.

Firdousi describes how Alexander, when governor of Persia, used oil fires for fusing building materials, and there are repeated references to flaming arrows which undoubtedly imply the use of oil.

The oil at Bakou is frequently mentioned from A.D. 272 onwards, and in 1574 it was noted that the oil from this place was transported widely. Apparently it was during the early part of the eighteenth century that the Hindus erected a temple at Sourakhani over a gas seep. After various changes Russia regained Bakou in 1806, but the bulk of the oil was still exported to Persia. In 1819 the output was estimated at 4000 tonnes. Oil was also exploited primitively at Masdjid-i-Sulaiman, Naft-i-Safid, etc.

In 1884 Hotz obtained a concession from the Persian Government and put down some shallow pits. His deeper well was badly placed. The exploration rights changed hands several times without success being achieved, and in 1901 they were acquired by D'Arcy. After costly and fruitless work at Tchiab-Sourkh in 1902-1904, he sought aid which resulted in the formation of the Concessions Syndicate in 1905. When almost at the end of its resources, the latter drilled a well at Maidan-i-Naftun which struck a large oil reservoir at 360 m., and was the first of a series of prolific producers. In 1909 the Anglo-Persian Oil Co., Ltd., was formed.

Bitumen which was used for preserving mummies was undoubtedly the source of the word mummy in Persian. The bitumen was highly esteemed, being collected with great ceremony, and was believed to have great curative properties. G. D. H.



BOOK REVIEW.

Standard Methods for Testing Tar and Its Products. Second Edition. 1938. Pp. 434.
Standardization of Tar Products Tests Committee. 166, Piccadilly, London, W.1.
Price 21s. net.

The first issue of these methods was in 1929, following a decision reached at a Tar Conference held in Manchester some two years after the first issue of the Institute of Petroleum Technologists' "Standard Methods for Petroleum Products" (1924). There can be little doubt but that the example of the petroleum industry brought home to the tar industry the real necessity for a similar effort. The tar distillation industry had been in existence many years prior to the petroleum industry, and in different laboratories, some small, some large, quite crude experimental methods, incapable of giving anything but approximate results, had been practised. The old retort distillation test for crude benzole, with the bulb of the thermometer in the liquid, and an arbitrary "stop point" at 120° C. is a good example.

Within the last twenty years there has also been a change in the requirements of many of the products; for all practical purposes road tar was a new material, and motor benzole was soon found to require methods of test which had no significance in the old days of benzene as a raw material for the dyestuff and other "chemical" industries—the "gum" tests, for example.

Methods of test obviously do not call for critical comments or any discussion of detail, seeing that they are all sponsored by Committees and Panels composed of those in daily touch with the methods. As with the I.P. standardization work (with which there has been a happy working contact), revision of the First Edition (1929) has been carried out through a very similar organization, certain tests omitted, others added, with considerable increase in size, the Second Edition running to over 400 pages.

Almost from the commencement the main Committee and the Revisionary Panel have been fortunate in having as Chairman Mr. H. W. James, and as Hon. Sec. Mr. R. M. Drake. Whilst the many members of the various Committees and Panels have done yeoman service, the writer feels confident all will agree that the energy, experience and tact of the Chairman and Hon. Secretary have been of the utmost value in ensuring the production of a Second Edition which is a fine example of team work in this important industry.

J. S. S. BRAME.

BOOKS AND PUBLICATIONS RECEIVED.

Report of the Fuel Research Board for the Year Ended 31st March, 1938. Pp. x + 255.
H.M. Stationery Office, London. Price 4s.

Further work is reported on the synthesis of lubricating oils from the gases produced in the total gasification of coal. The spirits produced by the process have been found after secondary treatment to possess good characteristics in regard to coke numbers, both before and after oxidation, setting point and flash point, whilst the other properties are up to the average values for mineral oils. The absolute viscosity range does not quite lie within that suitable for a winter grade oil, but it is thought that this could be remedied in future preparations by distilling off a smaller fraction of the less viscous constituents.

Experimental work has been put in hand to determine whether the high rates of wear encountered in coal-dust diesel engines can be reduced to practicable proportions by suitable choice of material for the liner, piston and rings. The results so far obtained are promising.

British Standard Specification No. 143, December, 1938, for Malleable Cast Iron and Cast Copper Alloy Pipe Fittings for Steam, Water, Gas and Oil. Pp. 58. British Standards Institution, 28, Victoria Street, London, S.W.1. Price 3s. 6d.

This specification deals with the dimensions of malleable cast iron and cast copper alloy pipe fittings, but is not concerned with the composition of the material. The fittings are, in general, suitable for working pressures up to 200 lb. per sq. in. in the case of water and up to 150 lb. per sq. in. in the case of steam, gas or oil.

INSTITUTE NOTES.

FEBRUARY 1939.

FORTHCOMING MEETINGS.

Tuesday, 14th March, 1939, at the Royal Society of Arts, John Street, London, W.C. 2. **Symposium on "Dangerous Gases in the Petroleum and Allied Industries."**

Morning Session, 10.30 a.m.—12.30 p.m. A series of papers dealing with **"The Detection and Inflammability of Dangerous Gases."**

Afternoon Session, 2.30 p.m.—5.0 p.m. A series of Papers dealing with **"The Toxic Effects of Gases, and Safety Measures, including Safety Measures for Electrical Equipment."**

Evening Session, 6.15 p.m.—7.30 p.m. Lecture by Dr. R. B. Vallender : **"The Detection of Toxic Gases."**

22nd—24th May, 1939. Summer Meeting of the Institute at Birmingham.

NORTHERN BRANCH.

Thursday, 9th March, 1939, at 7.15 p.m., at the Engineers' Club, Albert Square, Manchester. **Annual General Meeting** and **"The Search for Oil in Great Britain,"** by G. W. Lepper, B.Sc., A.R.C.S.

Branch Lecture.

Friday, 10th March, 1939, at 7.0 p.m., at the University, Edmund Street, Birmingham. **"The Search for Oil in Great Britain,"** by G. W. Lepper, B.Sc., A.R.C.S.

SCOTTISH BRANCH.

Friday, 24th March, 1939. **"The Planning of a Modern Colliery,"** by William Reid, B.Sc., Full particulars can be obtained from Prof. W. M. Cumming, D.Sc., Royal Technical College, Glasgow.

STUDENTS' SECTION (LONDON BRANCH).

Thursday, 16th March, 1939, at 5.45 p.m., at the Offices of the Institute, The Adelphi, London, W.C. 2. **"The Application of Science to the Petroleum Industry,"** by G. H. Coxon.

TRANSFERS TO NEW CLASSES OF MEMBERSHIP.

The Temporary Regulations relating to the transfer of the existing members to the new classes of membership, as set out in the leaflet sent to all members of the Institute, were approved at the Special General Meeting held on 10th January, 1939.

Members who wish to transfer to one of the new classes of membership are requested to submit their applications as early as possible on the forms provided for the purpose at the back of the leaflet convening the Special General Meeting.

NEW MEMBERS.

The following elections were made by the Council in accordance with the By-Laws, Section IV, Para. 7, at the Council Meeting held on Tuesday, 14th February, 1939.

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

Members.

DRAPER, Philip	London.
HULTGREN, Axel I.	Argentine.
MYHILL, Walter Edward	Roumania.
STEHRMAN, Carlyle John	Ohio.

Associate Members.

HILL, Dennis Stanley	London.
HORNE, Donald	Chesterfield.
NEALE-MAY, William Montague	S. Africa.
NERCESSIAN, Vahram	Iraq.
REID, William	Scotland.
SEMMENS, Graham Corkill	Trinidad.
TURNER, Bernard Evelyn Boileau	London.

Students.

BALDWIN, Alan Henry	Birmingham.
BARBER, Bernard Thomas	Birmingham.
BEAUMONT, Geoffrey Keith	Birmingham.
BROWN, Thomas	Birmingham.
BRUIJN, Jan Antonie de	Holland.
BRUNNER, Donald George	Birmingham.
DAFT, Victor Donovan	Birmingham.
DAVIS, Alec Kenneth	Birmingham.
DIXON, John Frederick	Birmingham.
DOWN, Arthur Lewis	London.
GEORGE, Henry George	Birmingham.
GREGSON, Hayse Dennis	Birmingham.
HEKMAT, Hossein Khan	London.
HOWARD, Frederick George	Birmingham.
IRELAND, Gilbert Robert	Birmingham.
JU, Ting Y.	Birmingham.
LAUCKNER, Frank Godfrey	Birmingham.
LONGBOTTOM, Frank William	Birmingham.
LUCIE-SMITH, Geoffrey Ross	Birmingham.
MOORE, John Lawson	London.
NAGHAVI, Said	Birmingham.
OWEN, Peter Benedict	Birmingham.
PERKS, John Gilbert	Birmingham.
WILLIAMS, Harold Robert	Birmingham.
WILLMOTT, Ivor Alan	Birmingham.

CANDIDATES FOR ADMISSION.

The following have applied for admission to the Institute or transfer to another grade of membership, and in accordance with the By-Laws the proposals will not be considered until the lapse of at least one month subsequent to the issue of this *Journal*, during which any Member or Associate Member may communicate by letter to the Secretary, for the confidential information of the Council, any particulars he may possess respecting the qualifications or suitability of any candidate.

The object of this information is to assist the Council in grading candidates according to the class of membership.

The names of the candidate's proposer and seconder are given in parentheses.

ALLEN, Samuel, B.Sc., Chemical Engineer (*Anglo-Iranian Oil Company*), 162, Purley Downs Road, Sanderstead, Surrey. (*C. V. Hill ; A. E. Dunstan.*)
ANNAN, John Greig, B.Sc., F.I.C., Works Manager and Chief Chemist, Scottish Oils, Ltd, Grangemouth, Stirlingshire. (*R. Crichton ; G. H. Smith.*)

- ANNAN, John Greig, jr., B.Sc., Chemist (*Scottish Oils, Ltd.*), 207, Boness Road, Grangemouth, Stirlingshire. (*R. Crichton; G. H. Smith.*)
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Honorary Secretary.

PERMANENT COUNCIL OF WORLD PETROLEUM CONGRESSES.

Meeting at Brussels, 28th January, 1939.

The Permanent Council of World Petroleum Congresses met at Brussels on Saturday, 28th January, 1939, under the Presidency of Col. Louis Pineau. Two new members were co-opted to the Permanent Council: Sr. Puppini, representative of Italian petroleum organisations; and the President of the Organizing Committee of the 3rd World Petroleum Congress due to be held in Berlin in June, 1940.

The next meeting of the Council has been arranged for Düsseldorf at the beginning of March. This meeting will be concerned with the question of general organisation of the 3rd Congress.

PERSONAL NOTES.

- Mr. R. K. DICKIE is home from Iran.
- Mr. G. HESELDIN has returned to Iraq.
- Mr. G. E. HIGGINS has left for Venezuela.
- Mr. C. E. KEEP has returned to Burma.
- Mr. A. N. LUCIE-SMITH has returned to Venezuela.
- Mr. H. L. LYNE will shortly return home from Sarawak.
- Mr. F. A. MARR has returned from India.
- Mr. C. B. ROACH is in the U.S.A.

Correspondence or *Journals* forwarded to the following members have been returned, and the Secretary would be pleased to receive any information regarding their present address: E. C. BROWN, K. BURTON, M. CAPPER, O. C. ELVINS, V. C. S. GEORGESCU, J. J. L. HAMILTON, J. R. HORTH, A. D. JONES, J. LANDER, H. R. LOVELY, I. LUSTY, F. MACKLEY, A. MACLEAN, G. P. MELVILLE, C. A. MOON, S. NICOL, N. D. ROTHON, H. G. SPEARPOINT, J. TAVANA, and A. H. WILLIAMS.

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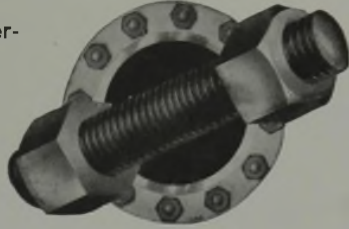
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British India.—A. Couret, DIGBOI, Assam.

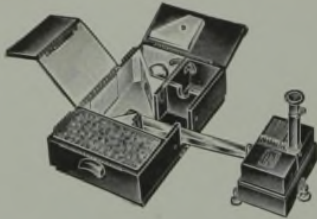
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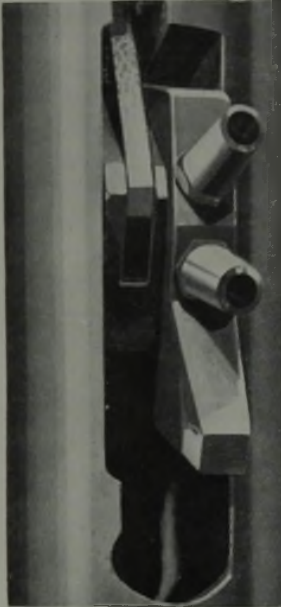
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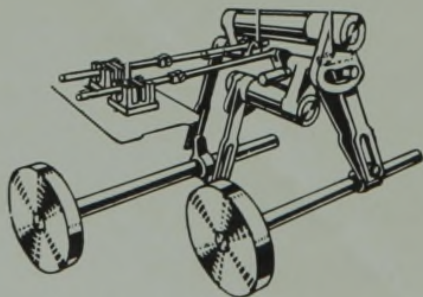
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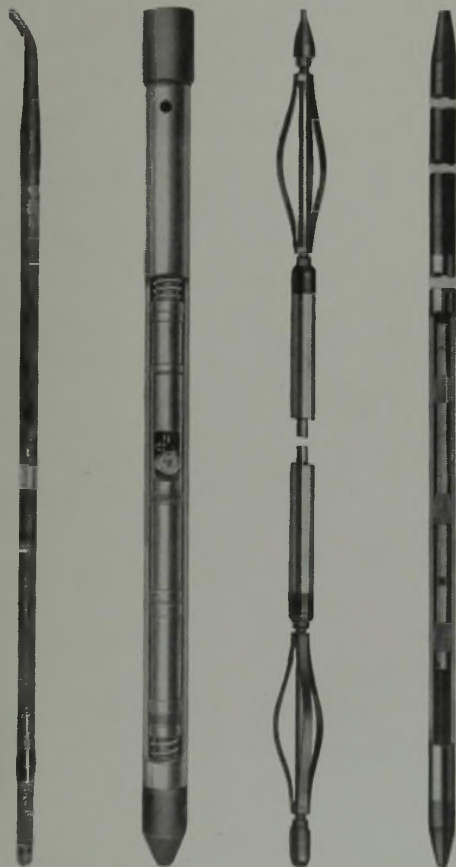
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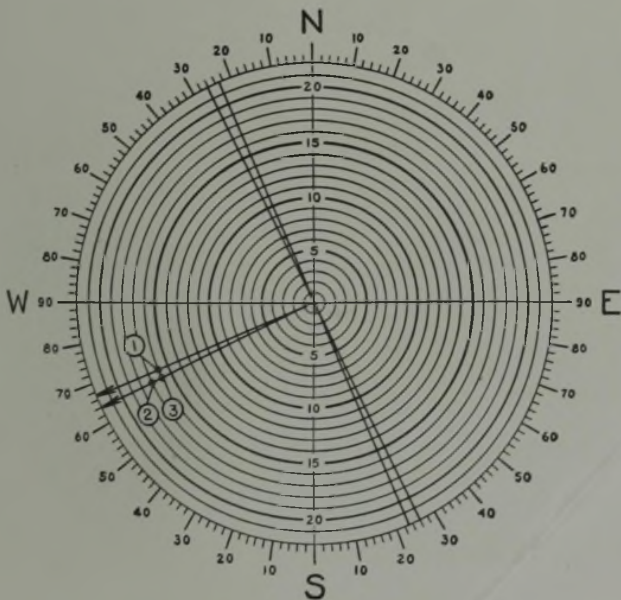
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