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THE MANUFACTURE AND TESTING OF ROOFING-FELTS AND DAMP-COURSES.*

By D. M. WILSON, M.C., B.Sc., A.I.C.

INTRODUCTION.

LITTLE is known about the early use of roofing-felt, but it appears that about 150 years ago, Dr. Faxé, a Swedish naval adviser, nailed paper to a boarded roof, coated it with hot wood-tar, and finished it with coloured mineral carths.

In 1844 ships' sheathing-paper was dipped into a melted mixture of pine-tar and pitch, the excess squeezed out, and the surface sprinkled with sand. At a later date the paper in the form of rolls was run through saturators containing heated tar.

The roofing-felt and the asphalt industries seem to have developed simultaneously, and in Europe both received a stimulus from their use in the waterproofing of magazines and fortifications. Owing to faults in the early days, the use of mastic asphalt in Germany was generally restricted to the covering of horizontal roofs and those with a small slope, so the arched surfaces of bridges and tunnels were proofed with laminated felt imported from England. This appears to have been the material known as "sheathing" felt, made from flax waste and manufactured by David Anderson in 1849. The name was derived from its use as an underlay to the copper plates of ships. Manufacture in Germany began about 1857. Although in France and England mastic asphalt was at first the more favoured waterproofing medium, in Germany the use of felt rapidly outstripped that of asphalt, because tar for impregnation was available in most cities, whereas the asphalt rock had to be carried considerable distances.

It was not until many years after the invention of sheathing felt that it became popular as a roof-covering. When used for this purpose it was coated, directly after laying, with a mixture of pitch and tar, and then sprinkled with sand; with periodical dressings of tar it provided an economical and satisfactory form of roof-covering. Considerable quantities were supplied during the Crimean War, and the War Office continued to use this type of felt up to the end of 1914, when supplies of flax and jute became scarce. Its durability was remarkable, and some of the felt supplied during the Crimean War was still in service at Shorncliffe camp in 1918. Thus a felt originally used for damp-proofing found a use for roofing.

Mention has been made of the early use of paper coated with tar following the crude method of coating brown paper *in situ* with tar; developments along this line followed, chiefly in America. A soft sheet of fibre made on a paper-making machine was treated with tar and sold in rolls. This material had the advantage that it did not require any coating treatment when first laid on the roof. At a later date asphaltic bitumen

* Paper presented to a meeting of the Asphaltic Bitumen Group of the Institute of Petroleum held in London, on Tuesday, 16th April, 1940.

came into use. Although asphaltic bitumen was introduced into Germany from America soon after 1890, tar maintained its dominant position in the roofing-felt industry there until a few years ago. In this country stearine, water-gas, oil-gas, and coal-tar pitches were used for many years before they were replaced by asphaltic bitumen.

Many types of roofing-felt are manufactured to-day, and will be considered under the names given in the British Standard Classification for Bituminous Roofing Felts, no. 747. Similarly, damp-courses will be referred to under the headings given in Part 2, B.S.S. 743. Actually there is really no hard-and-fast division; roofing-felts are quite frequently used for damp-courses, and damp-coursing felts have been used for roofing. Both can be made on the same machines and from the same materials.

BITUMEN FELTS.

Roofing-felts of this type all consist of a saturated fibre base which may be given a coating either on one side only, or on both.

Fibre Base.

The fibre base, sometimes known as the "rag felt," or the "dry felt," is prepared on a paper-making machine from selected rags containing a large proportion of wool, cotton, and linen, together with jute and Manila fibres. Sometimes a proportion of wood pulp is introduced. The base is made in different thicknesses, ranging usually from about 20 to 80 mils, according to the thickness of the bitumen felt it is desired to make. The composition of the base is of considerable importance; the larger the proportion of wool, cotton, and linen, the better will be the felt. Wool fibres are the most desirable, and cotton and linen come next in durability, whilst jute fibres deteriorate more rapidly. Fillers should not be employed, as their presence would affect the porosity of the felt.

The amount of saturant that the base will hold in its fibres and voids depends largely on its composition and on the skill of the maker, and can be expressed as the weight of bitumen carried by 100 grams of the felt base. In practice the "percentage saturation" may vary from 100 to 180. The waterproofing qualities of the finished felt depend on the presence of a high percentage of bitumen, so the higher the percentage saturation of the base, the better.

Although the fibre base should be very porous, it must nevertheless have sufficient tensile strength to be handled on the saturating- and coating-machines without breaking; the strength is considerably increased by saturation, as shown in Table I.

Asbestos felt may also be used as the fibre base for roofing and damp-coursing felts, and is made in a similar way to rag felt. It usually contains about 85 per cent. of asbestos fibre, and cow-hair is often added to increase the strength and the percentage saturation. Typical properties in the unsaturated and saturated condition are also indicated in Table I.

The asbestos in this example is the thinner by 10 mils, but although its tensile strength is lower in the unsaturated condition, it is considerably higher when saturated. It is saturated and coated in exactly the same manner and on the same machine as the fibre-base felts.

TABLE I.

Type of base.	Thick-ness of felt, mils.	Satur-ation, %.	Tensile strength at 20° C. — lbs. per 1 inch wide; test-piece 2 inches wide, 7 inches between grips, load applied at rate of 65 lb. per minute.			
			Dry-fibre base.		Saturated-fibre base.	
			Cut lengthwise.	crosswise.	lengthwise.	crosswise.
Rag felt	50	139	15	13	37	29
Asbestos felt.	40	87	6	4	51	41

Saturant.

The saturant for the fibre base is usually an asphaltic bitumen of the straight-run type having a penetration at 25° C. of between 100 and 200, and it calls for no particular comment. The harder the saturant the greater will be the tensile strength of the saturated fibre; if it is too hard, however, the pliability of the finished felt will be affected.

Coating.

A coating of asphaltic bitumen is applied to one or both sides of the saturated fibre to act as a seal and fill any voids in the fibre that may remain after saturation. The coating is usually a blown bitumen, and some of the roofing-felt manufacturers prepare their own blown grades, in which the properties are adjusted according to the country to which the felt is being supplied.

A high melting point is necessary to withstand exposure to high sun temperatures, but at the same time the felt must remain pliable over a wide range of temperature. If the coating is too hard it is liable to crack while being laid, and moisture may gain admittance to the fibre base. A blown bitumen has a higher melting point for a given penetration than straight-run material, and is more pliable over a range of temperature. This fact is indicated in Table II, where 10 represents a high and 1 a low degree of pliability, the tests being made on strips of bitumen $\frac{1}{16}$ -in. thick.

TABLE II.

Type of bitumen.	Melting point (R. & B.). ° C.	Penetra-tion at 25° C.	Penetra-tion index.	Pliability.										
				Temp. of test, ° C.										
				25.	20.	15.	10.	5.	4.	3.	2.	1.	0.	
Blown X . . .	85.5	34	+ 4	10	10	10	10	10	10	10	10	10	10	10
Blown Y . . .	82.0	18	+ 2	10	10	10	10	6	4	4	4	4	4	4
Straight-run . .	61.1	24	- 0.5	10	10	10	10	1	1	1	1	1	1	1

A column has been included to show the penetration index* of the bitumens. This method of indicating the temperature susceptibility is most useful, and the table also indicates that for practically the same

* Pfoiffer and Van Doormal, *J. Instn Petrol. Tech.*, 1936, 22, 414.

melting point the bitumen with a high penetration index is more pliable over a range of temperature than the one having a lower index.

Blowing of Bitumen.

The properties of a blown bitumen can be varied according to the method of blowing employed and the "stock" from which the bitumen is blown.

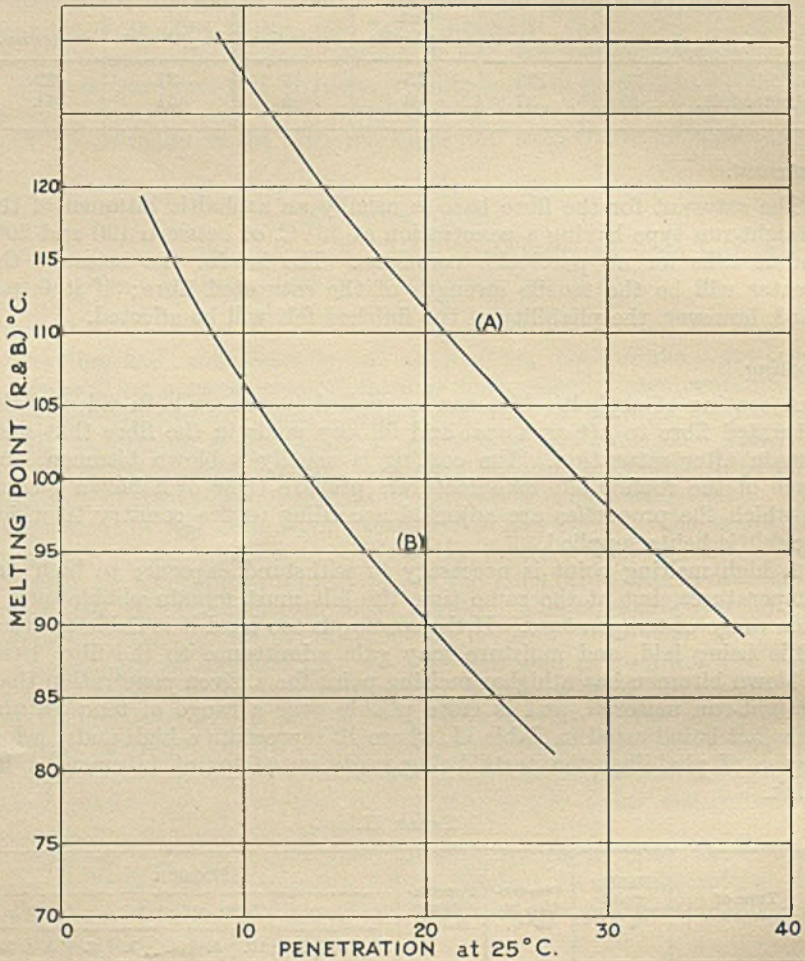


FIG. 1.

CURVES SHOWING MELTING POINT/PENETRATION RELATIONSHIP OF BITUMEN BLOWN (A) UNDER PRESSURE AND (B) BY NORMAL METHOD.

In order to obtain both a high melting point and a high penetration, the blowing stock must be in the nature of a very heavy asphaltic oil, and the process should be conducted so that the maximum oxidizing effect is obtained with the minimum loss of light oils. One method of obtaining such conditions is to blow the bitumen in a partly closed con-

tainer under pressure. The air is introduced at a pressure of 50-60 lb. per square inch, the outlet to the container is partly closed, and the volume of air is adjusted so that the pressure in the vessel is 20-30 lb. per square inch. Fig. 1 shows the effect on the penetration/melting-point curve when the same material—a very heavy fuel oil—is blown under pressure and when no pressure is applied, all other conditions being kept constant. The application of pressure leads to the production of a bitumen with a higher penetration index, as the loss of distillate is reduced. When heavier material, such as 500 penetration bitumen, is used as the blowing stock, a blown bitumen results which has a lower penetration index.

A bitumen with a high penetration index may also be obtained by fluxing a bitumen blown to a high melting point—say, 135° C.—with some of the distillate produced in the blowing process, or with a flux oil. One disadvantage, however, of some of the high-penetration-index blown bitumens is that they may suffer a considerable loss when heated to, say, 200° C. Table III shows the loss on heating undergone by two blown bitumens of practically the same melting point, but different penetration indices. The bitumen with the high penetration index shows a much higher loss on heating than the one with the lower index.

TABLE III.

Blown bitumen.	Melting point (R. & B.). ° C.	Penetration at 25° C.	Penetration index.	Loss on heating 5 hrs. at 200° C. %
Type 1 . . .	80.5	35	+ 3.5	2.3
Type 2 . . .	80.0	23	+ 2.5	0.5

It is often necessary to compromise and use a bitumen with a lower penetration rather than run the risk of a high loss in subsequent heating processes. "Sweating" of the light oils from the finished felt may occur with a bitumen of high penetration index and tend to discolour the talc or other dressing. Judgment has to be employed, therefore, in choosing bitumens of suitable properties. In a general way it may be said that for this country the most suitable coating is a blown bitumen having a melting point of about 85° C. with a penetration at 25° C. between 20 and 30. Most manufacturers have drawn up their own formulæ after many years of experience, various additions being made to the blown bitumen to improve its weathering properties and pliability.

Dusting and Finishing Materials.

The dusting-powder is talc, mica, sand, or other suitable powder, which is spread on the surface of the coated felt as it passes through the machine. The object of the dusting is to prevent the felt from sticking to the rollers of the machine and when in the rolled condition. It also increases the thermal reflectivity when the felt is laid on a roof. Care must be taken not to apply too heavy a dressing, as it may present difficulties when the felt is being laid. The dusting compound should be removed as far as possible when two surfaces are being stuck together, as on a built-up

roof, or it will interfere with the adhesion of the bitumen used to cement the layers.

Various-coloured grits are also applied to the finished surface of some types of felt instead of a dusting-powder, and are lightly pressed into position by rollers while the bitumen coating is still warm. These grits protect the bitumen from the ageing effect of sunlight, particularly if they are light in colour, and at the same time improve the appearance of a roof. The grits may either be coloured naturally, or in some of the brighter colours may consist of a slate or granite coloured artificially.

Manufacture of Bitumen Felts.

A modern machine is shown diagrammatically in Fig. 2. The fibre base is received in rolls, and is unwound on to a "Looper" (A), which

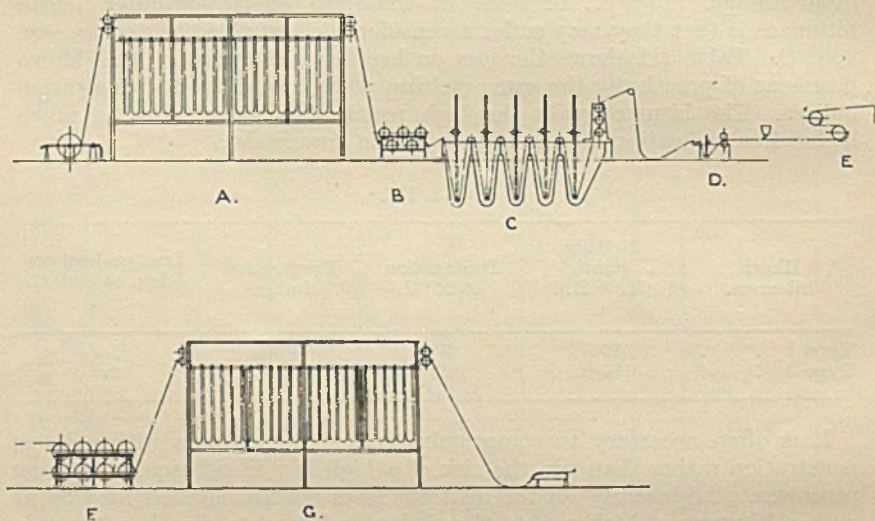


FIG. 2.

DIAGRAM OF FELT SATURATING AND COATING MACHINE.

builds up a reserve so that the machine need not be stopped to splice on a new roll of fibre. The fibre passes over drying-cylinders (B) and then through the saturator (C). It passes to the coating-rolls (D) on to the first cooling-rollers (E), where the dusting material is applied, and then to further cooling-rollers (F) and another looper (G), where it is finally cooled and stored before reeling. The tanks holding the saturant and coating may be jacketed and heated by the circulation of hot oil from a central heater such as that indicated in Fig. 3, which is operated by gas burners (Fig. 4). The temperature of the oil on leaving the heater is maintained automatically at 295° C. The jackets of the bitumen supply-pipes from the storage-tanks to the machines are also heated by hot oil from the circulator.

The machine (Fig. 5) will make all the types of felt detailed in B.S.S. 747, viz.—

1 A. *Impregnated Bitumen Felts.*—These, more often referred to as

“saturated” felts, merely consist of the fibre base impregnated with bitumen at a temperature of 150–200° C. Any moisture remaining in the fibre is expelled and evaporated from the surface of the saturant. Any excess of saturant on the surface of the fibre is removed by passing the felt through heated rollers, and it is then cooled and rolled.

1 B. *Sanded Bitumen Felts*.—A somewhat harder saturant is employed than in the previous type, and some is allowed to remain on the surface. Sand is used as the finishing material. Although still employed abroad, the method is practically obsolete in this country.

1 C. *Self-finished Bitumen Felts*.—The saturated fibre is passed into the hot coating mixture and then through a pair of steam-heated rollers, where the coating is spread evenly over the surface of the saturated fibre and bonds with the bitumen saturant. The thickness of the coating is regulated by the distance the two rollers are set apart. The felt may be given a “veined” finish by allowing the rollers to rotate, or may be smooth finished if the top roller is kept stationary while the felt is pulled through; the underside is smoothed by applying a scraper-blade to the felt after passing over the bottom roller. After dusting, the felt is cooled on rotating cylinders (Fig. 6) through which water is circulated.

1 D. *Mineralized Bitumen Felt*.—In this class the top side of the felt is finished with coloured grit. A 2-inch margin of the felt may be left free from granules so that a lap can be made during laying. The manufacturing process is largely the same as for the self-finished felts, except that the felt passes through additional press-rolls to ensure that the grit is well bedded. For satisfactory results attention must be paid to the grading of the grit; it should all be retained on an 18-mesh B.S.I. sieve and completely pass the 8-mesh.

1 E. *Reinforced Bitumen Felts*.—The strength of a self-finished felt is considerably increased by adding a layer of hessian. The fibre base and the hessian are passed through the coating process together and become firmly bonded. The felt is used for many purposes, particularly as a sarking under slates.

IMPREGNATED FLAX AND HAIR FELTS.

Although this type of felt is not now so extensively employed for roofing purposes, on account of the necessity for periodical dressing, it is still used as an underlay for slates, tiles, and asphalt, and acts as a thermal insulator. It also insulates the asphalt from any movements of the underlying structure.

The base consists of cow-hair or long-fibred flax and jute waste, or mixtures of all these materials, which are carded and thoroughly mixed in a machine similar to that used in the preparation of cotton for spinning; all the dust is extracted, and the fibres remain. The fibres are passed through the preparing-machines, in which they are separated by intimate contact with a rapidly rotating striking-cylinder, the surface of which is fitted at about 2-inch centres with stout iron pins. The fibres are delivered to a rotating perforated cylinder under suction, which extracts the dust and delivers the fibres as a soft “batt” to a flat table,

where it is carried forward to the actual felt-machine for impregnation. It then passes through two rollers, which remove the surplus saturant. Whilst still warm the saturated felt is treated with fine dust derived from flax waste (termed "shives," and consisting mainly of the straw fibre of flax), to prevent the laps from adhering in the rolls. The felt then passes through another series of rollers to compress it further and smooth the surface, and is finally rolled up.

The felt was originally made to suit the width of the Muntz metal plates used for wooden ships. The plates were 32 inches by 20 inches and the felt was cut into pieces 32 inches by 40 inches, each sheet thus being sufficient for laying under two of the metal plates. The felt is still manufactured in a 32-inch width, as are all felts in Part 2 of B.S.S. 747. These are:—

2 A. *Impregnated Flax Felt, Black.*—The base consists of flax and jute and the saturant is a fluxed coal-tar pitch.

2 B. *Impregnated Flax Felt, Brown.*—Felts in this class are generally described as inodorous, and are intended for use where the smell of coal-tar pitch is not desired. The base is the same as in class 2 A, but they are impregnated with mixtures containing wood tars and pitches, together with resins.

Similar brown and black felts are also made in which animal hair forms the batt instead of flax and jute, and are classified under the headings, 2 C and 2 D.

DAMP-COURSING FELTS.

From the earliest days of the industry, bituminous felts found a use in the damp-coursing of buildings and bridges. At first they were made from flax felt, strips being cut across the width of the roll, giving a length of 32 inches. These strips, after dipping into a pan of hot tar, were placed on a pile of sand; additional sand was sprinkled on top, and then another length was placed on top of the first. When the workman had prepared his strips in this manner, he proceeded to lay them on the brick-work. About 30 years ago the modern type came into use. It consists of a fairly thick bitumen coating on a hessian or fibre base. Sheet lead is also introduced to give added protection against the percolation of water in wet situations.

The components are the same as for roofing-felts, and the same type of bitumen is used as the saturant for the hessian or fibre base. Blown bitumens generally form a considerable part of the coating, and the penetration index may with advantage be higher than in the case of roofing-felts, in order to maintain the pliability of the somewhat thicker coating. This is usually given greater stability by the addition of a filler, such as slate dust, limestone, asbestos, or asbestine. The melting point of the coating can be adjusted by the addition of fluxing oils to suit widely differing conditions, and satisfactory results have been obtained both in the tropics and in the coldest Northern climates.

Attention must be paid to the pliability of the coating to avoid cracking when a roll is laid in cold weather, although it is true that with suitable coatings the cracks are self-healing when under load in a wall. The

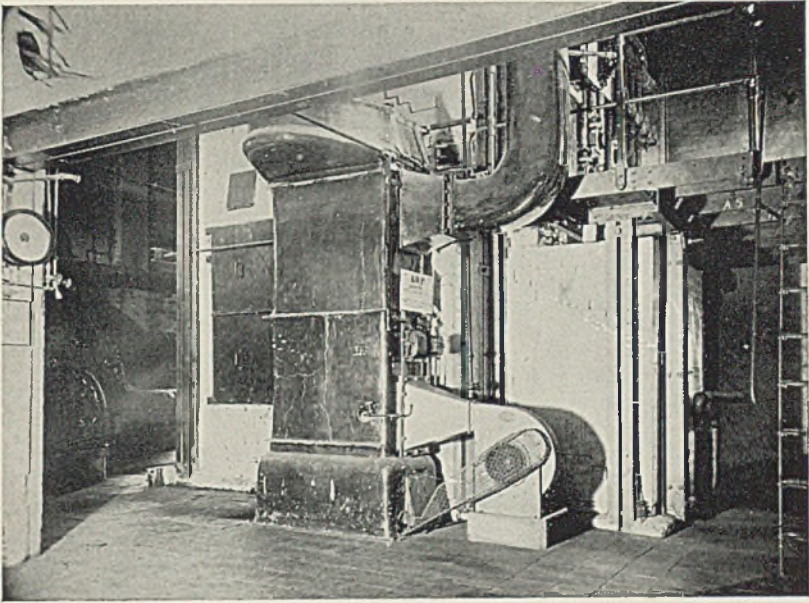


FIG. 3.
CENTRAL OIL-HEATING INSTALLATION.

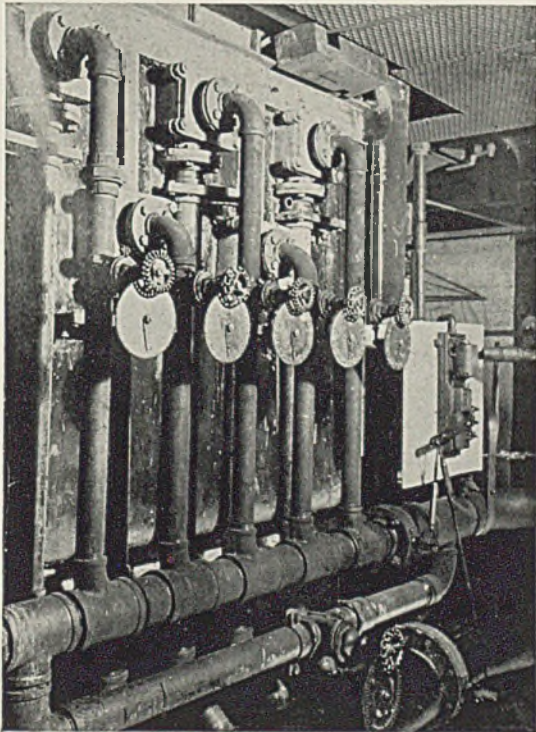


FIG. 4.
GAS BURNERS OF OIL HEATER.

[To face p. 384.]

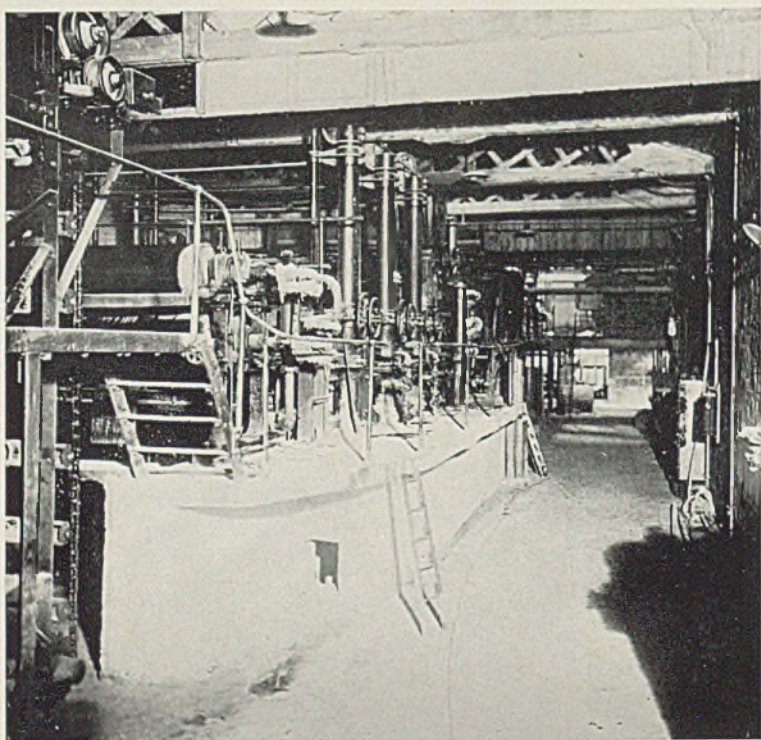


FIG. 5.

BITUMEN FELT MACHINE.

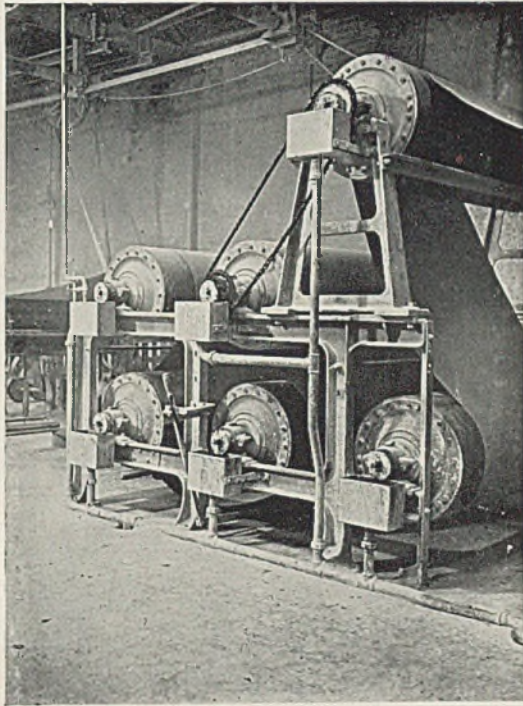


FIG. 6.
COOLING ROLLS.

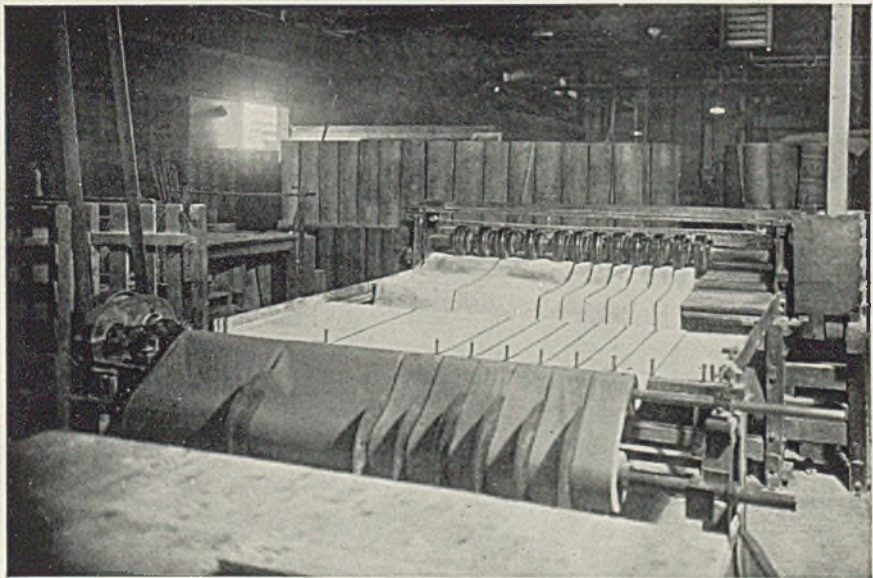


FIG. 7.
CUTTING MACHINE FOR DAMPCOURSING.

coating must also be sufficiently stable, or there would be a risk of its partial extrusion when laid.

Damp-course felts are generally made in 3 or 6 feet widths, which are cut to the width required by a machine of the type shown in Fig. 7 and are generally sold in rolls 8 yards long.

Bituminous damp-courses are classified in B.S.S. 743 according to the base materials, viz. :—

- Hessian base.
- Fibre base.
- Lead core and hessian base.
- Lead core and fibre base.

Many other types are also on the market in which different combinations of the base materials are employed. As in the case of roofing, asbestos felt may replace the fibre base.

TESTING OF ROOFING-FELTS AND DAMP-COURSES.

A large number of tests can be applied to roofing-felts, but standardization is difficult, as with all products containing bitumen, and it is not easy to eliminate the personal factor. Fortunately the manufacture of roofing-felts is largely in the hands of firms of repute, some of whom have been in the business for the best part of a century, and they can be relied upon to maintain a high standard.

Tests on Raw Materials.

Fibre Base.—This is tested for thickness by a Leunig micrometer gauge, which exerts a uniform pressure at its contact surfaces, for Mullen strength and for its weight per square yard. Several tests have been described for measuring the saturating capacity, but it is far more satisfactory to saturate a length of the fibre on a machine and then extract a known weight of the saturated material to determine its bitumen content.

Bitumen.—Control of the bitumen is both by melting point and penetration, and rapid methods are preferable. For the melting point test a multiple apparatus * has proved valuable, and for the penetration test the method † that enables a figure to be obtained in 30 minutes has now been used for many years.

Pitch and Tar.—The consistency of the grades used for saturating felt can usefully be controlled by the Float test (A.S.T.M. D.139-27) at a temperature of 75° C.

Finished Bitumen Felt.

Pliability.—One of the most important tests that can be applied to a roofing-felt is that for pliability. A method has been standardized by the A.S.T.M. in which the strips of felt, 6 inches × 1 inch, after immersion in water at an appropriate temperature, are immediately bent through

* *Chemistry and Industry*, 1938, 33, 767.

† *Ibid.*, 1932, 10, 204.

180° at a uniform speed in exactly 2 seconds around a mandrel. The pliability can be expressed by a number, thus :—

1. Cracks entirely through the sheet on the 25-mm. mandrel.
2. Cracks part of the way through the sheet on the 25-mm. mandrel.
3. Cracks on the 25-mm. mandrel.
4. Cracks on the 20-mm. mandrel.
5. Cracks on the 15-mm. mandrel.
6. Cracks on the 10-mm. mandrel.
7. Cracks on the 5-mm. mandrel.
8. Cracks when bent through 180° over a $\frac{1}{16}$ -inch (1.588-mm.) mandrel.
9. May be bent through 180° over a $\frac{1}{16}$ -inch (1.588-mm.) mandrel in one direction without cracking, but will crack when bent through 360° in the opposite direction.
10. May be bent through 180° over a $\frac{1}{16}$ -inch (1.588-mm.) mandrel in one direction, and then through 360° over a $\frac{1}{16}$ -inch mandrel in the opposite direction without cracking.

By making the tests at temperatures ranging from 25° C. downwards, a very good " picture " can be obtained of the pliability of the felt. The thickness of the film has a considerable effect on the results, and in Table IV figures are given for layers of bitumen prepared by pouring the melted material on to an iron plate, treated with dextrin solution to prevent sticking, to the approximate thickness required. The thickness is finally adjusted by passing the bitumen sheet when cold through a pair of rollers.

TABLE IV.

Thick- ness, in.	Pliability. Temp. of test, ° C.											
	25.	20.	15.	10.	5.	4.	3.	2.	1.	0.	-2.	-4.
$\frac{1}{32}$	10	10	10	10	10	10	10	10	9	8	6	1
$\frac{1}{16}$	10	10	10	10	10	10	9	9	9	9	5	1
$\frac{1}{8}$	10	10	10	10	5	1	1	1	1	1	1	1

It is obvious, therefore, that the thickness of the coating on a felt must be taken into account when considering the results of pliability tests. More consistent results could no doubt be obtained if the bending of the felt round the mandrel could be done mechanically, but nevertheless in its present form the test provides useful information.

Tensile Tests.—In one Government specification the tensile strength is required to be tested on strips of felt 2 inches wide by 7 inches between grips, cut from the roll both in the direction of its longer length and cross-wise, the rate of loading being 65 lb. per minute. The strength is expressed as the load in lb. per inch width required to break the test-piece.

A lever-machine fitted with grips of the type used for textile testing can be employed, the load being applied by either lead shot or water run into a canister hung from the end of the beam.

The test-pieces should be aged for at least one week in the case of newly

manufactured felt. Temperature has a considerable effect on the results, as shown in Table V, and should be quoted with the strength figures. The elongation at the breaking point can also usefully be recorded.

TABLE V.

Tensile strength :— Load in lb. per inch width required to break test-pieces.	Conditioning of test-piece.	
	Exposure for 6 hrs. in atmosphere of 65% R.H. at temp. of 21° C. (70° F.).	Exposure for 6 hrs. in atmosphere of 65% R.H. at temp. of 11° C. (52° F.).
Lengthwise	27	35
Crosswise	20	26

Weathering Tests.—The weathering qualities of a felt may be gauged by making pliability and tensile strength tests on strips that have been exposed in some type of artificial weathering apparatus, such as the Fugitometer.

Surface changes as the felt undergoes ageing may also be recorded by making reflectivity or colour tests on the surface and then repeating the tests at intervals as ageing proceeds. A photo-electric colorimeter may be used for this purpose, a portion of the original surface which has not been exposed being used as the reference standard. In this way changes in the surface characteristics which affect the reflectivity may be recorded as a single number, and the rate of change can give a useful indication of the extent to which the felt will resist the effects of weathering.

Tests on Coating.—Application of the bitumen recovery methods to a felt will only give the properties of the mixture of the coating and the saturant. It is often possible, however, to remove surplus dusting material and then scrape off sufficient of the coating unmixed with saturant for a melting point and penetration test.

Examination of "Dry Felt."—The bitumen may be removed by solvents in a Soxhlet extractor, so that the fibre base may be examined for thickness and weight per square yard. By measuring the thickness of the felt before and after extraction, the thickness of the coating may be determined.

Finished Flax and Hair Felts.—The approximate content of saturant is determined in a Soxhlet extractor using benzene, a thin wad of cotton wool being placed in the side-tube to act as a filter in preventing hairs from passing through. If tar is present a considerable portion of the free carbon comes over with the liquid. After extraction the hair is dried and teased out on a sieve to separate it as far as possible from the free carbon. The weight of the carbon is added to that of the extracted tar, which is obtained after evaporating the benzene and drying to constant weight at 105° C.

The percentage of animal hair may be determined by extracting a weighed amount of the "batt" with 2 per cent. caustic soda. By washing and drying the residue the amount of hair dissolved out can be determined.

Damp-courses.

The same tests are applied as in the case of roofing-felts. In addition, the resistance to the percolation of water may be tested by clamping a test-piece between two flanges, one of them being screwed on to a length of 3-inch pipe supported vertically. The pipe is filled with water and the damp-course can be submitted to the pressure of any desired head of water.

Conclusion.

These briefly are the methods of test in use in the industry at the present time, and suffice for the routine control of production. There are many other possible methods of distinguishing between a good felt and one that is not so good, and they are being explored.

The author's thanks are due to the Directors of Messrs. D. Anderson & Son, Ltd., Stretford, for permission to publish this paper.

DISCUSSION.

The CHAIRMAN (Mr. J. S. JACKSON) commented on the fact that the paper was particularly welcome, as it was probably the first paper on this aspect of the subject given in this country. He was pleased to note that in a modern plant for the manufacture of roofing felts special care was taken to dry the felt before it entered the impregnating bath. If damp felt were used, frothing frequently occurred. It was not always appreciated that quite small quantities of water of the order of 0.02 per cent. would cause asphaltic bitumen to froth, and such quantities could easily be introduced by using damp felt.

He asked the author whether he had experienced any special difficulties due to foaming caused by aeration.

He expressed surprise that manufacturers of roofing felt should still carry out their own "blowing" operations, since the blowing of relatively small quantities of bitumen must be a very inefficient and costly operation.

He referred to the difficulties experienced by the trade in connection with the staining of felts as the result of "sweating," and inquired whether the author had arrived at any satisfactory explanation of this trouble.

He noted that "filled" blown bitumens were used for the coating of damp-courses, and he was interested to know whether the use of lightly blown bitumens filled with a fibrous material had been tried for coating ordinary roofing felts.

In connection with the testing of roofing felts, he suggested that too much importance should not be attached to superficial colour changes resulting from the exposure of felts during the weathering test.

He suggested that a further paper dealing with the use of roofing felt would serve a very useful purpose.

He felt that the use of this excellent material was not widely understood, and its possibilities were certainly not generally appreciated.

Mr. A. V. HUSSEY, as a humble user of roofing felt, suggested that one of the chief weaknesses was its lack of resistance to tearing. Reference had been made to the employment of hessian for the fibre base, and it seemed to him that the incorporation of such a material must inevitably greatly improve the felt, particularly with regard to resistance to tear. In connection with the testing of felts, it occurred to him that the application of the Mullen test, as used for paper, would serve as a valuable index of the quality of a roofing felt. Furthermore, it was a well-known fact that, for example, four or five layers of paper were more resistant to shock and other stresses than the same thickness of paper but not made up of five plies. By analogy, therefore, it would seem that roofing felt made with two or more fibre bases interspersed with bitumen would be stronger than one fibre base covered with increasing thickness of bitumen according to quality and weight.

Reference had been made during the discussion to the possible deterioration and decomposition of vegetable fibre when embedded in bitumen, resulting in damage to the bitumen coating itself. Mr. Hussey was strongly of opinion that no such dissociation or decomposition was likely to occur unless it arose from some interaction between the bitumen or constituents of the bitumen and fibre itself. He quoted cases where cellulosic materials had been removed from ancient buildings after having been embedded in calcareous clay for more than 2000 years without deterioration.

Lastly, in connection with the laying of felt, he did not think it was generally appreciated how important it was that certain preliminary precautions should be taken to avoid the creasing, folding, and bulging which were generally noticeable on roofs covered with roofing felt. An expert linoleum layer would not attempt to fix the linoleum in position until the expiration of two or three or more weeks after unrolling, whereas in the case of roofing felt it was customary to undo the roll and immediately fix on the roof. In such circumstances it was not likely that satisfactory results would be secured while the unevenness of the material permitted of the penetration of weather, draughts, and high winds which ultimately lead to tearing and other damage.

In his reply, the AUTHOR said that no difficulties arose on account of frothing unless the fibre base had not been dried properly. Some manufacturers found it useful to blow their own bitumen, as it enabled them to produce grades which were not normally on the market. "Sweating" often seemed to be due to the use of a bitumen with too high a penetration index for a particular climate: it appeared that there was a tendency for oils to separate from the bitumen, and so cause staining.

Fibrous fillers such as asbestos and asbestine were often used in the coating for roofing felts as well as dampcourses.

Replying to Mr. Hussey, he said that it was only the very light grades of felt that lacked resistance to tearing, and the grades were only intended for use on small temporary buildings. Heavier grades with a comparatively high tensile strength were available for more permanent work.

Felts reinforced with hessian were finding extensive uses in connection with A.R.P. purposes and for the screening of roof lights. If a sheet of this reinforced felt was stuck to the glass, the latter might be shattered by an explosion, but the felt would prevent the pieces from dropping on the workers below. It would also continue to screen the factory lights and enable production to continue.

The Mullen test was used extensively in the testing of the fibre base, but a tensile machine of the type employed for textiles was better for dealing with the finished article.

Roofing felts with more than one layer of fibre had been made for many years, and were preferred in certain countries.

Finally the author agreed that great care should be exercised in the laying of the felt, but that was a subject almost big enough for a separate paper. Most manufacturers recommended that felt should be unrolled, cut to length, and laid out for as long a period as possible before laying on the roof.

STUDIES ON THE SEPARATION OF PARAFFIN WAXES. PART I. PRINCIPLES INVOLVED IN THE FRACTIONAL MELTING OF WAX.*

By M. F. SAWYER, Ph.D., A.M.Inst.Pet., T. G. HUNTER, D.Sc.,
M.Inst.Pet., and A. W. NASH, M.Sc., F.Inst.Pet.

In recent years considerable research has been conducted in the Department of Oil Engineering and Refining, the University of Birmingham, on phase-rule studies of systems important to the petroleum industry. The more recent publications have dealt with dewaxing¹ and double-solvent refining,² each subject having been investigated on a quantitative fundamental basis. Further research was commenced with the object of applying established principles to a study of the refining of waxes, the ultimate intention being to apply the results obtained to the sweating and solvent-refining processes employed industrially in the production of refined paraffin wax.

In many investigations—both qualitative and quantitative—on lubricating oil, a physical property such as V.G.C. has been employed successfully to indicate the quality of the oil. V.G.C. cannot be applied to waxes for this purpose, and the selection of a suitable physical property of wax which is characteristic is a matter of considerable difficulty. This paper, which is of an introductory nature, therefore includes a discussion of the choice of a physical property to characterize the wax.

FUNDAMENTAL BASIS OF THE INVESTIGATION—THE NATURE OF PARAFFIN WAX.

In any study involving phase transformations the initial consideration must be that of the nature of the substance. Attempts have been made in many researches to elucidate the composition of paraffin wax, and there is general support for the view that *n*-paraffins usually predominate. Hydrocarbons of at least one other type may also be present to some extent, probably dependent on the crude from which the wax is derived, but there is no general agreement as to whether these belong to the *iso*-paraffinic and/or naphthenic series. The waxes used in this study have constituents melting over the range 105–142° F. at least, and assuming that the components are *n*-paraffins, it is possible for those members to be present as shown in Table I.

Information concerning the state or nature of paraffin wax may be obtained from a consideration of the behaviour of wax during melting.

A pure substance is characterized by a sharp melting point, this being the temperature at which, on heating, the entire mass melts, or, on cooling, the entire mass solidifies. Paraffin wax, however, does not possess a sharp melting point, and the melting or solidification process takes place over a temperature range. It was first suggested by Gurwitsch³ that the

* Paper received April 1940.

single *n*-paraffins are partly isomorphous in relation to each other, and that during the solidification of paraffin wax, solid solutions and not chemically pure compounds, separate. In the case of non-polar organic compounds three conditions must normally be satisfied before two substances can form solid solutions:—

- (1) the chemical constitutions must be similar;
- (2) the crystal structures must be similar;
- (3) the molecular volumes must be nearly equal.

TABLE I.

<i>n</i> -Paraffin.		M.W.	M.Pt. (° C.).	M.Pt. (° F.).	B.P. under 15 mm. ° C.
<i>n</i> -Heneicosano	C ₂₁ H ₄₄	296·3	40·4	104·7	215
<i>n</i> -Docosane	C ₂₂ H ₄₆	310·4	44·5	112·1	224
<i>n</i> -Tricosano	C ₂₃ H ₄₈	324·4	47·4	115·3	234
<i>n</i> -Tetracosane	C ₂₄ H ₅₀	338·4	51·1	124·0	243
<i>n</i> -Pentacosano	C ₂₅ H ₅₂	352·4	53·3	127·9	259
<i>n</i> -Hexacosane	C ₂₆ H ₅₄	366·4	56·6	133·9	262
<i>n</i> -Heptacosano	C ₂₇ H ₅₆	380·4	59·5	139·1	270
<i>n</i> -Octacosano	C ₂₈ H ₅₈	394·4	61·6	142·9	278

Applying these conditions to the *n*-paraffins included in Table I, it is reasonable to suggest, for example, that there is a greater possibility of solid solution formation between *n*-C₂₈H₅₈ and *n*-C₂₇H₅₆ than between *n*-C₂₈H₅₈ and *n*-C₂₁H₄₄. Assuming, then, that the paraffin wax consists in the main of members of the *n*-paraffin series, it is probable that on solidification the components would not all simultaneously solidify to form one solid solution, but that different solid solutions would successively separate—the first to be formed consisting of two or more of the higher components.

Except for the indirect evidence of Gault and Boisselet,⁴ who stated that the melting point of a mixture of waxes lies between the melting points of the components, the idea of solid solution formation remained unconfirmed by experiment until the work of Myers and Stegeman,⁵ who investigated the equilibria existing in mixtures of waxes of different melting points. By repeated carefully controlled fractional distillations under 0·1 mm. pressure of a large quantity of wax, and the ultimate collection of fractions boiling over narrow ranges, waxes of different melting points were obtained. The liquid–solid equilibrium curves were determined for blends of two of these close-cut fractions *A* and *B* over the complete range 0 per cent. *A*–100 per cent. *B*, and the nature of the diagram obtained provides evidence that wax mixtures form a continuous series of solid solutions. This view was confirmed by a similar series of experiments with two other close-cut wax fractions.

More recently, Verdonk⁶ has studied the melting curves of pure *n*-paraffins and commercial waxes, and suggests that the behaviour of the commercial waxes may be explained as being that of a mixture of solid solutions of substances which have two enantiotropically related modifications.

The original theoretical ideas of Gurwitsch have therefore received

experimental support, and any attempt to construct an equilibrium diagram applicable to the fractional melting of waxes could reasonably be developed from the basis indicated by the consideration that paraffin wax consists of a series of solid solutions—probably with *n*-paraffins predominating. Various types of solid-liquid equilibrium diagrams are described in the literature, but in view of the observation of Gault and Boisselet, and the findings of Myers and Stegeman, to which reference has been made, any such diagrams applicable to the process of fractional melting of waxes are more likely to resemble those for a continuous series

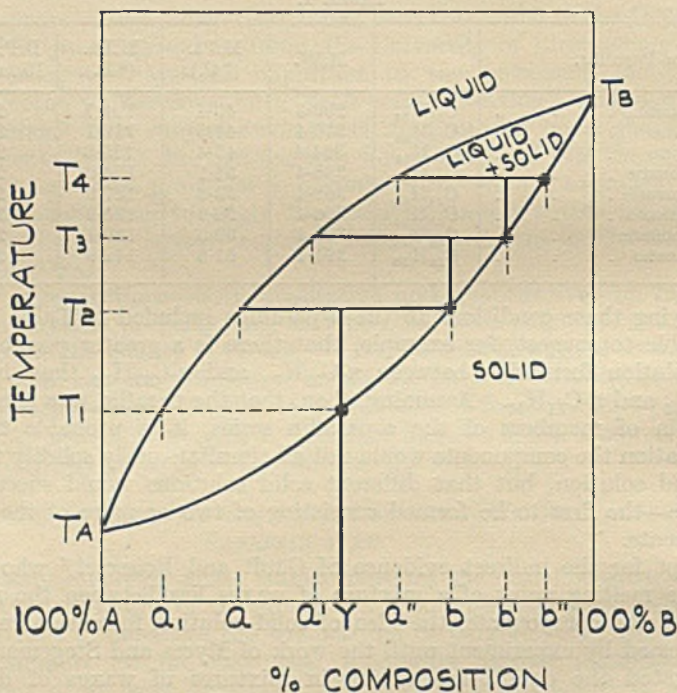


FIG. 1.

of solid solutions rather than those in which solid solutions of minimum or maximum melting point are formed. It is necessary, therefore, to make an elementary and brief study of a typical equilibrium diagram of this nature.

CONTINUOUS SERIES OF SOLID SOLUTIONS—TYPICAL EQUILIBRIUM DIAGRAM.

Fig. 1 is a typical diagram for two substances *A* and *B* which melt respectively at temperatures T_A and T_B . The upper curve is the "liquidus" or equilibrium curve of the liquid solution, and the lower is the "solidus" or equilibrium curve of the solid solution formed by these substances. Any system of composition represented by a point in the

region above the liquidus curve is entirely liquid, and by a point in the region below the solidus curve is entirely solid. Any system of composition represented by a point in the region between the solidus and the liquidus curves is partly liquid and partly solid.

Consider, now, the effect of slowly heating a mixture of composition Y . No phase change occurs until the temperature T_1 is reached, at which stage liquid of composition a_1 appears. At T_2 liquid of composition a co-exists in equilibrium with solid of composition b , and it may readily be shown that the ratio

$$\text{solid : liquid} = aY : Yb.$$

Let the liquid phase be completely removed at the temperature T_2 and let the remaining solid phase attain equilibrium at temperature T_3 , where the liquid phase is of composition a^1 and the solid phase is of composition b^1 . Again completely remove the liquid phase and let the remaining solid phase reach equilibrium at T_4 , where the compositions of liquid and solid phases are a'' and b'' , respectively.

Thus, in this particular three-stage removal of liquid phase the composition of the solid phase changes from Y to b'' and the percentage yield of b'' may be calculated as indicated above.

If, therefore, solid of composition Y is taken at temperature T_1 and the temperature is increased by means of an infinitely large number of infinitesimally small increments, and the resulting liquid phase is removed from the solid after each temperature increase, it follows that the composition of the solid present at any stage will change from Y to 100 per cent. B along the lower curve $T_A T_B$. In this ideal process pure B , the higher-melting component, will therefore ultimately be obtained.

MODIFIED REPRESENTATION OF EQUILIBRIUM DIAGRAM FOR A CONTINUOUS SERIES OF SOLID SOLUTIONS.

Fig. 1 represents the usual and simplest form of the equilibrium diagram for two substances forming a continuous series of solid solutions. It is clear, however, that it may also be represented in the modified form shown in Fig. 2, where composition is characterized by a physical property, in this case freezing point. In this diagram, equilibrium temperature is plotted against freezing point, defined in this case as that temperature at which solid first appears on cooling a liquid containing any proportion of components A and B . It is clear from Fig. 1, that as the property of freezing point does not follow the simple mixture rule, the curve relating freezing point to composition is an essential feature of Fig. 2, in order that computations may be evolved in a manner similar to that indicated for the previous figure.

The liquidus curve is a 45° line, as the freezing point of the liquid phase formed by any mixture is the same as the equilibrium temperature (Fig. 1). The solidus curve in Fig. 2 may easily be obtained by re-plotting the data from Fig. 1.

It follows that if a mixture of the two components, of freezing point T_s , and therefore composition C_s , is allowed to reach equilibrium at a temperature T and then separated completely into the two phases, the liquid phase will be of freezing point T_1 and composition C_1 , whilst the

solid phase will be of freezing point T_2 and composition C_2 . Also the ratio

$$\frac{\text{Liquid phase}}{\text{Solid phase}} = \frac{C_2 - C_s}{C_s - C_1}$$

If freezing point in any particular case followed the simple mixture law, it is obvious that for purposes of computation the lower curve (Fig. 2) showing freezing point *versus* composition would be unnecessary, as the relative amounts of the liquid and solid phases would be given by $(T_2 - T_s)$ and $(T_s - T_1)$ in Fig. 2, respectively. In such a case, the upper curve of Fig. 1 would have been a straight line.

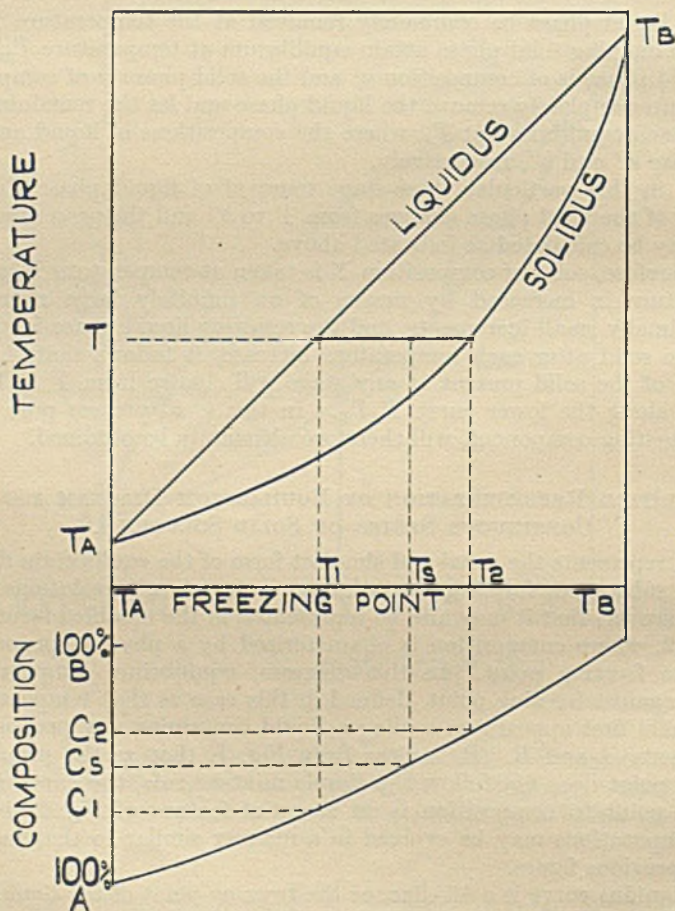


FIG. 2.

DETERMINATION OF EQUILIBRIUM DIAGRAM.

Two general methods may be employed to determine the equilibrium diagram given by substances forming solid solutions:—

(1) *Thermal Analysis*.—A method based on the determination of melting and solidification points for mixtures of known composition:

(a) from the study of heating or cooling curves; or (b) by direct observation of the melting range; and

(2) *Analysis of Equilibrium Mixtures*.—Allowing a suitable mixture to attain equilibrium at a temperature within the melting range of the components, separating the liquid from the solid phase, and then analysing the two phases.

The first method was used by Myers and Stegeman in the experiments already mentioned, but these workers were dealing with two very close-cut fractions, each melting over a range of not more than 1°C ., and which were treated as if they were pure compounds. The actual determination of the melting and solidification points of known mixtures was therefore possible. This method cannot, however, be applied to mixtures of waxes containing unknown quantities of many components, and it is therefore necessary to resort to method (2) above, but even this latter method is not free from experimental difficulty.

Paraffin wax has a high retentive power for hydrocarbon liquids—*e.g.*, oil—and if the last traces of such liquids are to be removed from the wax, special precautions must be taken. It might be anticipated then that solid wax would possess a high retentive power for liquid wax, and that complete separation of the two wax phases formed in the equilibrium experiments would not be easily achieved.

PHYSICAL PROPERTY FOR THE CHARACTERIZATION OF WAX PHASES.

The unknown composition of the wax presents an immediate difficulty, and it is therefore necessary to employ some physical property—*e.g.*, molecular weight, melting point, etc.—to characterize the liquid and solid phases formed in the equilibrium experiments in the same way as freezing point has been used to characterize composition in Fig. 2. The essentials of the physical property to be used for this purpose are:—

(1) For maximum convenience it should follow the simple mixture rule—*i.e.*, the physical property of a blend of two waxes should be a linear function of the percentage of the higher (or lower) of the two component waxes, in order that the equilibrium diagram shall apply quantitatively without the use of a secondary curve as in Fig. 2. If a secondary curve has to be employed in the manner illustrated in Fig. 2, then this curve must show the property selected as a function of composition or as a function of another physical property which follows the mixture rule.

(2) It should be capable of accurate determination as the melting range of the wax stock employed is limited, and hence change of physical property with changing composition will be small.

(3) It should be related preferably to the melting point, as this is, from the industrial viewpoint, the most important property of a wax.

As commercially refined paraffin wax contains more than two components, any physical property will be merely an *average* property, and will not, in general, give an indication of the exact composition of the wax. If, for example, three normal paraffins P_1 , P_2 , P_3 , have molecular

weights (in increasing order) of M_1 , M_2 , M_3 , respectively, then there is a very large number of possible blends each having a molecular weight of M , where M is somewhere within the range M_1 to M_3 . Since a single physical property is insufficient to define the exact composition of mixtures containing more than two components, any equilibrium diagram for paraffin wax making use of one physical property only will probably have limited applications. This point will be dealt with in detail later.

The physical properties which might possibly be used are as follows :—

- (a) molecular weight ;
- (b) refractive index ;
- (c) specific gravity ;
- (d) aniline point ;
- (e) melting point.

These properties will now be considered in greater detail.

(a) *Molecular Weight*.—Molecular weight, of course, follows the simple mixture rule, but is difficult to determine accurately in the case of waxes. Using even the ebullioscopic apparatus of Menzies,⁷ and extrapolating the molecular weight values to infinite dilution, it is difficult to achieve an accuracy of ± 2 per cent. consistently.

Considering this limit of accuracy with reference to *n*-docosane and *n*-octacosane—two *n*-paraffins the melting points of which are within the wax range

	M.W.	M.Pt. (° F.).
<i>n</i> -Docosane	310.4	112.1
<i>n</i> -Octacosane	394.4	142.9

it is observed from these values that the melting point is raised by 30.8° F. for an increase of 84 units in molecular weight.

The melting point is therefore affected to the extent of 0.37° F. for each unit increase in molecular weight. Assuming that the molecular weight may be estimated with an accuracy of ± 1 per cent.—a figure attained only with considerable difficulty in practice—the experimental error on a molecular weight of 300 would be ± 3 units, equivalent to ± 1.1 ° F. With a probable error of such magnitude in the determination, molecular weight is less suitable than melting point for use in the construction of the equilibrium diagram where accuracy to within ± 0.1 ° F. is desirable and may be obtained in the melting-point determination. The assumption that the increase in molecular weight per unit increase in melting point for paraffin waxes is approximately equal to the corresponding increase for the two *n*-paraffins considered is justified, since the molecular weight/melting-point curve of the commercial waxes used in this work was found to coincide fairly well with the corresponding curve for *n*-paraffins of molecular weight within the wax range. In this connection it may be noted that Berne-Allen and Work⁸ also found close agreement between the experimental values of the molecular weights of the waxes they used and those for the *n*-paraffins of corresponding melting points.

(b) *Refractive Index*.—Refractive index is a property which may be determined rapidly and accurately. It is especially convenient, since only a very small quantity of material is needed for the determination. For waxes of melting point lower than 60° C., the refractive index of the

liquid sample is normally determined at 60° C. For waxes of higher melting point the refractive index may be measured at a temperature 1° C. above the melting point and then reduced to 60° C. by applying a correction of 0.0004°/C. Using an Abbé Refractometer, the refractive index may be estimated to ± 0.0001 , and the following are typical values for fractions collected in laboratory "sweatings" of an oil-free wax stock.

TABLE II.

M.Pt. (° F.).	n_D^{60}
112.6	1.4328
119.4	1.4334
127.1	1.4345
137.3	1.4362
142.7	1.4373

These figures indicate that the value of n_D^{60} increases by approximately 0.00015 for each 1° F. increase in melting point, and as n_D^{60} may be estimated to ± 0.0001 , the possible error in terms of melting point is more than $\pm 0.6^\circ$ F.—an amount which favours the use of melting point rather than refractive index in this connection.

(c) *Specific Gravity*.—Figures given in the literature for the melting point/specific gravity of wax are very variable, but the following data given by Morris and Adkins⁹ for American wax may be cited to give an indication of the relation between the two properties :—

TABLE III.

M.Pt. (° F.).	d_{60}^{130} F.
103	0.778
109	0.777
115	0.780
118	0.782

It is clear that the specific gravity varies only slightly with melting point, and assuming even that the values for the waxes of 115° and 118° melting point are typical, it is observed that a difference of 3° F. affects the specific gravity by only 0.002. The specific gravity of a wax is not normally required to more than ± 0.001 , and as it is doubtful whether the value may be conveniently determined to an accuracy of ± 0.0002 , this property is not as accurate as melting point.

(d) *Aniline Point*.—The aniline point of wax may be readily determined, and individual readings do not normally differ by more than 0.2° C. It has been shown by Lord¹⁰ that there exists between the melting point and aniline point of commercial waxes a relationship which can be expressed by the equation

$$\text{Aniline Point (}^\circ\text{C.)} = 0.319 \times \text{Melting Point (}^\circ\text{F.)} + 75.6.$$

Thus, an increase of 0.319° C. in aniline point represents an increase of 1° F. in melting point, and assuming that aniline point may be determined

to $\pm 0.1^\circ \text{C}$., the possible experimental error would then be equivalent to $\pm 0.3^\circ \text{F}$. in melting point.

Aniline point is therefore the most promising of the properties so far reviewed, and is likely to lead to more accurate results than any other of these properties. The linear relationship between aniline point and melting point is, however, of special significance, as this means that if melting point does not exactly follow the mixture rule, then aniline point also behaves in the same way. Now a modification of the I.P.T. Setting Point Method (Serial Designation P.S. 11a) has been developed which may be used to determine melting point with an accuracy of $\pm 0.05^\circ \text{F}$., and it is clear, therefore, that no advantage is to be gained by using the property of aniline point in preference to melting point, especially as the latter is quite as easy to determine.

(c) *Melting Point.*—It was found that the melting point of a mixture of waxes could not be calculated accurately from the rule of mixtures. Blends of various waxes were found to have melting points slightly higher than the values calculated from the rule of mixtures. This was confirmed by the determination of the melting points (using I.P.T. Setting Point Method) for blends of two refined commercial waxes *H* and *L* of melting points 130.95°F . and 108.35°F ., respectively, over the complete range 100 per cent. *H* to 100 per cent. *L*. The actual melting point and that calculated from the rule of mixtures are given for each of the blends in Table IV below and are represented graphically in Fig. 3.

TABLE IV.

% <i>H</i> .	M.Pt. ° F. (actual).	M.Pt. ° F. (calculated).	Difference between actual and calculated.
100	130.95	—	—
90	129.15	128.7	+ 0.45
80	126.95	126.4	+ 0.55
70	124.9	124.2	+ 0.7
50	120.4	119.65	+ 0.75
40	118.0	117.4	+ 0.6
30	115.65	115.1	+ 0.45
15	112.1	111.75	+ 0.35

This is further illustrated by the following results given by Berne-Allen and Work for certain wax blends. The values calculated by the law of mixtures are included.

TABLE V.

Blend.	Actual M.Pt. (Berne-Allen).	Calculated.
Equal wts. of 52.8°C . and 64.4°C . M.Pt. waxes	59.1°C .	58.6°C .
Equal wts. of waxes of following M.Pts. 49.9, 52.8, 55.6, 60.3 and 64.4°C .	56.9°C .	56.6°C .

It will be noted that the experimental is slightly higher than the calculated value.

As would be expected, it is found that the smaller the difference between the melting points of the two stock waxes, the smaller is the deviation

between the actual and calculated values. For example, a 50/50 blend of two commercial waxes of melting points 130.95°F . and 119.85°F ., respectively, was found to have a melting point of 125.6°F ., compared with the calculated figure of 125.4°F .—a difference of 0.2°F .—whereas the difference between actual and calculated values for a 50/50 blend of 130.95°F . and 108.35°F . waxes is 0.75°F . (Table IV).

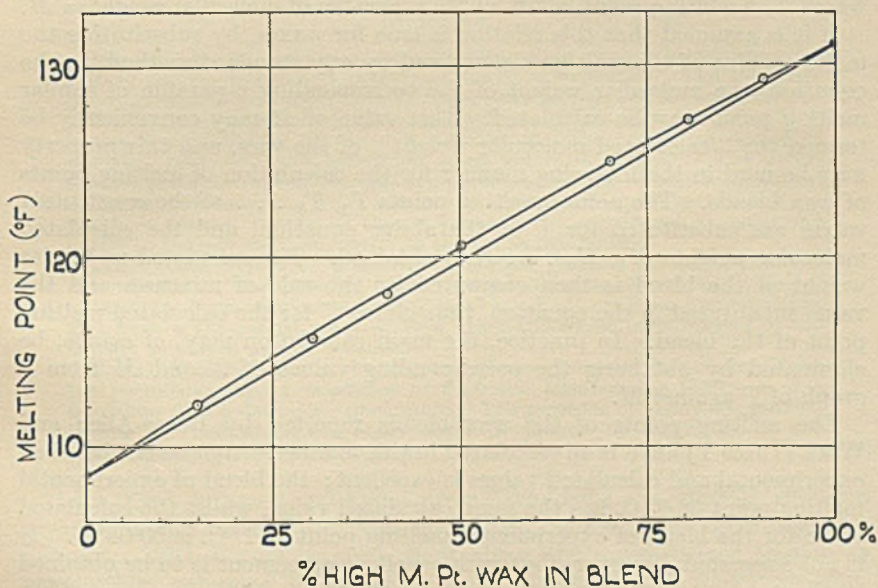


FIG. 3.

It is to be concluded that melting point is the most suitable physical property for the characterization of waxes, since :—

(a) it may be determined without difficulty to within $\pm 0.05^{\circ}\text{F}$., using a simple apparatus, and

(b) although not strictly following the mixture law, there is not a great difference between the experimental values and those calculated on the assumption that it does.

There is a further justification for its use, for although refined paraffin wax must meet definite minimum oil content and colour requirements, it is marketed primarily on the basis of melting point specifications.

MELTING POINT OF WAX AS A FUNCTION OF MOLECULAR WEIGHT.

Molecular weight strictly follows the mixture rule, and although, as already stated, its direct use is not considered suitable in this work, owing to the comparatively large experimental errors involved in its determination, the possibility of its indirect use was considered.

Now, the melting point/composition curve for mixtures of two waxes is similar in form to the melting point/molecular weight curve for the n -

paraffins within the wax range. It has been shown in a previous publication¹¹ that the relationship between molecular weight and melting point for these *n*-paraffins may be expressed as follows:—

$$T = \frac{414.5 M}{M + 94.4}$$

where *T* = melting point in °K of the *n*-paraffin of molecular weight = *M*.

If it is assumed that this relation is true for waxes, by substituting the melting point of the wax (as determined by any standard method) in the equation, the molecular weight of the corresponding *n*-paraffin of similar melting point may be calculated. This value of *M* may conveniently be termed the "calculated molecular weight" of the wax, and this property may be used in the following manner for the calculation of melting points of wax blends. The actual melting points *T*₁, *T*₂ . . . of the constituent waxes are substituted for *T* in the above equation, and the calculated molecular weights *M*₁, *M*₂ . . . thus obtained. The calculated molecular weight of the blend is then computed by the rule of mixtures and the value substituted in the equation, thus giving *T* for the calculated melting point of the blend. In practice, the main calculation may, of course, be eliminated by obtaining the corresponding values of *T* and *M* from a graph of *T* against *M*.

The melting points of the wax blends reported by Berne-Allen and Work (Table V) have been calculated in this manner. Agreement between experimental and calculated values is excellent; the blend of experimental melting point 56.9° C. has the same calculated value, whilst the calculated value for the blend of experimental melting point 59.1° C. is 59.05° C. It is not suggested, however, that such excellent agreement is to be obtained in all cases.

Assuming this relation to follow the mixture law, the yields of solid and liquid phases obtained in many equilibrium experiments (described in Part II) were calculated, and it was found that the agreement between experimental and calculated figures was, in general, closer than when melting point alone was used. The use of this relation for the wax experiment in this research is therefore justified.

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RESEARCHES ON ASPHALTENES. PART I.*

By F. J. NELLENSTEYN and J. P. KUIPERS.

SUMMARY.

The difference in solubility of asphaltic bitumen in solvents having the same surface tension (ether and petroleum spirit) is explained. The solubility curves of asphaltic bitumen from Trinidad-Lake asphalt and the separation of light-coloured compounds in the ether extract of this bitumen prove that incomplete miscibility of the oily medium and the protective bodies of asphaltic bitumens with petroleum spirit enters into action. The ether-soluble, petroleum-spirit-insoluble compounds appear to be present in almost all asphaltic bitumens. Their rôle in the asphaltic bitumen system has not yet been cleared up; they should probably be considered as belonging partly to the oily medium, partly to the protective bodies.

Ethyl ether should be preferred to normal gasoline for the asphaltene determination. The practical advantages of ether for the determination of asphaltenes have previously been pointed out.⁵ It appears, however, that also from a theoretical point of view ether should be used instead of the usually employed normal gasoline.

The other asphaltenes in the majority of cases are pure flocculates, whilst the petroleum-spirit precipitates of asphaltic bitumen are mixtures of a flocculate with a de-mixing precipitate. The presence of highly oxygenated bodies in Trinidad-Lake asphalt could be proved by extraction of the difference-asphaltenes with acetone.

ASPHALTIC bitumen extracted from Trinidad-Lake asphalt shows a great difference between the ethyl ether- and the petroleum ether-insoluble content. With a petroleum spirit of about the same surface tension as ethyl ether, values of 35-38 per cent., as compared with 20-24 per cent. for ethyl ether, are found. Although a quantitative relation between the surface tension of the flocculating agent and the amount of precipitate, as indicated by the surface-tension rule,¹ has been found² only for tar, generally also for asphaltic bitumen the lower surface-tension liquids cause a higher amount of precipitate. The great difference between the petroleum spirit and the ether precipitate therefore indicates an incomplete miscibility of the medium and the protective bodies of Trinidad asphaltic bitumen with petroleum spirit. In this case it is easy to prove that incomplete miscibility enters into action. The filtrate of the ether asphaltenes, after evaporation of the ether, gives with petroleum spirit a *rather light-coloured* precipitate.

Further, this incomplete miscibility of petroleum spirits may be deduced from the curves in Fig. 1. The precipitates are obtained in the following way :—

2 gm. of asphaltic bitumen are mixed in an Erlenmeyer with 100 mls. of the precipitating mixtures. The bitumen is divided into small pieces with a glass rod, and the Erlenmeyer left in a cool place ($\pm 5^\circ$ C.) overnight. The precipitate is filtered through a weighed Schleicher and Schüll filter no. 589,² and washed with 200 mls. of the precipitating liquid. When

* Paper received March 1940.

high-boiling liquids are used (petroleum spirit 80–100, 100–130 and their mixtures) the precipitate should be finally washed with 15 mls. pentane. The filter is dried, weighed, and burnt. The amount of precipitate is calculated from the difference in weight of the filter and the weight of the ashes.

The precipitating mixtures are composed of ether, various kinds of petroleum spirit, and mixtures of these liquids. The gasolines used have the following properties :—

	Specific gravity at 25°.	Surface tension dyne/cm. at 25°.
Petroleum spirit 40–60°	0.6482	17.5
" " 60–80°	0.6838	21.2
" " 80–100°	0.7085	22.7
" " 100–130°	0.7313	24.3

Table I shows the amount of precipitate which various mixtures give with asphaltic bitumen from Trinidad-Lake asphalt.

TABLE I.
Percentage of Precipitate.
Ethyl ether 20.0%.

Petroleum spirit, b.p.	Ethyl ether–petroleum spirit mixtures			100% petroleum spirit.
	3 : 1	1 : 1	1 : 3	
40–60°	22.3	24.2	27.8	38.0
60–80°	19.1	18.7	21.0	27.2
80–100°	17.5	17.4	19.9	25.9
100–130°	15.2	14.3	17.3	21.1

In previous publications,³ attention was directed to the fact that the combination of a flocculent with a partly miscible liquid of high surface tension leads to a solubility in the mixtures which may be higher than the solubility in both components separately. Conversely, as in the case of the curves in Fig. 1, one may conclude that incomplete miscibility of one of the components with the bitumen occurs if the concentration–insolubility curve shows a minimum or a marked deviation from the straight line connecting the values for the pure liquids (*AB*, *AC*, etc.). Here petroleum spirit is the incompletely miscible component.

From the foregoing it clearly appears that the great difference in solubility of asphaltic bitumen extracted from Trinidad Lake asphalt in ethyl ether and petroleum spirit of the same surface tension is in complete concordance with the surface-tension rule, the higher value for petroleum spirit being due to incomplete miscibility.

Little is known about the nature and amount of these petroleum-spirit-insoluble, ethyl-ether-soluble compounds, owing to the fact that in the ordinary asphaltene determination petroleum spirit is used as flocculent, and a mixture of the ether asphaltenes with these compounds is always

obtained. Therefore it seemed important to determine the amount of the ether-soluble, petroleum-spirit-insoluble compounds in various asphaltic bitumens. The following method has been used:—

The ether asphaltenes are precipitated according to the methods described on pages 401–2. The filtrate, after evaporation of the ether on a steam bath, is mixed with 100 mls. petroleum spirit (boiling point 40–60° C.). After standing over-night in a cool place ($\pm 5^\circ$ C.), the precipitate is filtered through a weighed Schleicher and Schüll filter no. 589² and washed with 200 mls. petroleum spirit. The filter is dried, weighed, and burnt. The amount of precipitate is calculated from the difference in weight of the filter and the weight of the ashes.

The result of these determinations is given in Table II.

TABLE II.

Asphaltic bitumen from	Method of manufacturing.	Softening point ring and ball, ° C.	Ether-asphaltenes, %.	Petroleum spirit 40–60 asphaltenes, %.	Petroleum spirit-ether-asphaltenes.	
					Calculated, %.	Determined, %.
Trinidad-Lake Asphalt.	Extracted with CS ₂	65.0	21.0	38.0	17.0	11.0
Boston asphalt						
(a) Kaboengka	"	105.0	38.2	47.4	9.2	6.7
(b) Wariti	"	84.0	25.3	38.0	13.6	8.7
Gilsonite	No treatment	—	41.6	62.2	20.6	7.2
Californian crude	Steam refined	49.0	6.1	17.6	11.5	10.7
Rumanian crude	"	55.0	17.3	25.3	8.0	6.8
South Mexican crude	"	57.0	23.1	20.1	6.0	4.7
Egyptian crude	"	53.1	22.6	34.6	12.0	8.0
Venezuelan crude	"	55.0	17.2	21.5	4.3	4.9
Dutch East-Indian crude	"	48.0	12.2	20.7	8.5	6.1
Mexican crude	"	118.0	39.3	47.0	7.7	6.1
" " " "	"	63.5	25.2	29.2	4.0	5.4
" " " "	"	56.0	22.4	27.8	5.4	4.1
" " " "	"	53.6	21.8	25.6	3.8	5.2
" " " "	"	47.6	20.8	24.8	4.0	6.3
Miri crude " " " "	"	52.5	1.3	5.7	4.4	5.5
Mesopotamia " " " "	No treatment	52.5	21.2	29.6	8.4	5.7
Venezuelan crude " " " "	Blown with air	57.8	21.6	25.4	3.8	0.0
Mexican crude " " " "	"	64.0	29.5	37.4	7.9	3.4
" " " " " " " "	"	175.0	48.2	55.6	7.4	1.5

It is evident that the presence in bitumens of compounds which are insoluble in petroleum spirit and soluble in ether is a generally occurring phenomenon certainly not restricted to the natural asphalt, although some of these—*e.g.*, Trinidad-Lake asphalt show a very high content of these bodies. Owing to this general occurrence it seems desirable to look for a name for these compounds. According to the Dutch nomenclature, all the flocculates and semi-flocculates of asphaltic bitumen are called asphaltenes; the name of the flocculent is to be added. So these ether-soluble, petroleum-spirit-insoluble compounds should be called (*petroleum-spirit 40/60-ether*) *asphaltenes* or, briefly, "*difference-asphaltenes*."

Table II demonstrates some striking facts. In the first place, the amount of difference-asphaltene is much more constant than that of the ether- or petroleum spirit-asphaltene. No correlation between the ether asphaltene content and the amount of difference-asphaltene exists. Asphaltic bitumens from the same crude (Mexican) with increasing softening

point show about the same amount of difference-asphaltenes, whilst the other asphaltenes increase from 20.8 to 39.3 per cent. Although these determinations are satisfactorily reproducible, it must be taken into consideration that the amount of precipitate depends greatly on the mode of operation.

These are conventional determinations; therefore a warning should be issued against going too far in reaching conclusions. The accuracy of the difference-asphaltenes determination is about ± 1 per cent., so that values of 4-6 per cent. should be considered as practically constant. The most important fact is the general occurrence of these difference-asphaltenes in asphaltic bitumens; the highest values are found for Trinidad-Lake asphalt

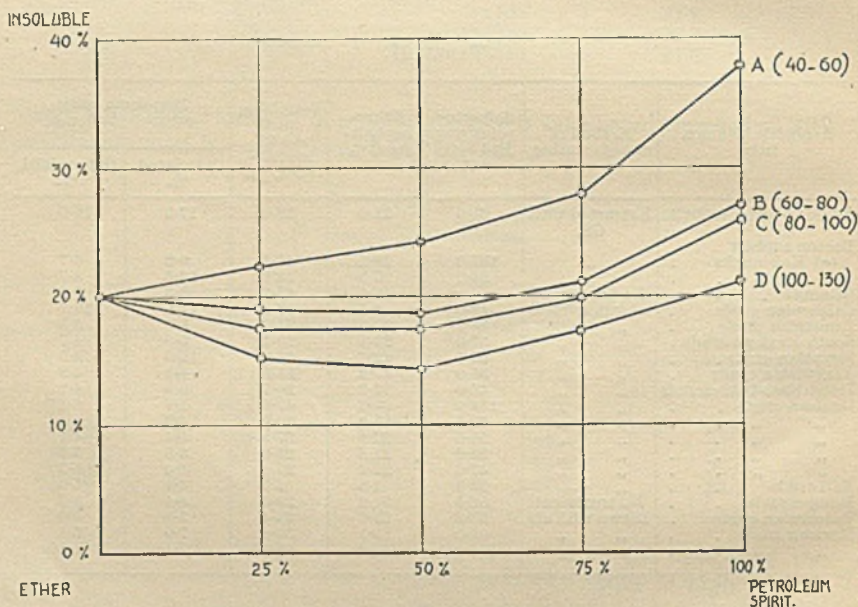


FIG. 1.

and Californian asphaltic bitumen, the lower values for the blown products. Miri asphaltic bitumen contains a normal percentage of difference-asphaltenes, but its ether asphaltene-content is low.

The calculated and determined values of the difference-asphaltenes are not the same; in most cases the determined values are lower. This may be caused by adsorption of the difference-asphaltenes by the micelle-kernels.

Generally the amount of ether asphaltenes is smaller than that of the flocculates of petroleum spirit with about the same surface tension. It is, however, possible, in connection with the fact that the nature of the compounds in asphaltic bitumen may vary considerably, that the ether-asphaltenes content will be found higher than that of the petroleum-spirit asphaltenes. We know only one such exceptional case; an asphaltic

bitumen from an Argentine crude with 17.2 per cent. ether- and 13.3 per cent. petroleum-spirit asphaltenes.

The rôle of the difference-asphaltenes is not yet cleared up; as a demixing product they may belong to the oily medium, but, on the other hand, they show peptizing properties for fine mineral powders. An analysis of a highly altered asphaltic bitumen (from the excavations at Mohenjo-Daro,⁴ Indus valley, 3000 B.C.) gave a similar indication. This 5000-years-old product had become partly insoluble in CS_2 ; the soluble part showed the following asphaltene content:—

	Per cent.
Ether asphaltenes	42.0
Petroleum spirit (40–60° C.) asphaltenes	67.7
Difference-asphaltenes	26.0

The high amount of difference-asphaltenes is an indication of the resistance of these bodies against alteration, but also of their peptizing properties; even the highly altered micelles may be peptized by the difference-asphaltenes. As peptizing compounds they belong to the protective bodies, and so to the micelle; but the fact that the difference-asphaltenes are easily separated from the micelles by ether proves that they belong at least partly to the oily medium.

The difference-asphaltenes from Trinidad Lake asphalt may be separated into an acetone-soluble part, a dark red amorphous body, which dissolves into acetone with a cherry-red colour, and a brown residue. On adding petroleum-spirit, the colour of the acetone solution changes into light brown; with more petroleum spirit a precipitate is obtained, the colour of which is lighter than that of the original product. An attempt to purify the acetone-soluble bodies by mixing with petroleum spirit to which some acetone had been added gave no result.

A lighter-coloured product could be obtained by distilling Trinidad-Lake asphalt in high vacuum and precipitating the distillate with petroleum spirit 40–60° C. The precipitate is washed with petroleum spirit; it dissolves in ether.

A comparison of the elementary composition of the ether asphaltenes, the difference-asphaltenes, the acetone-soluble compounds from the difference-asphaltenes and the petroleum-spirit insoluble compounds from Trinidad-Lake asphalt gives interesting indications regarding the nature of the difference-asphaltenes.

The ether asphaltenes obtained in the ordinary way from Trinidad-Lake asphalt still contain a considerable amount of ashes and ether. The product is purified in the following way:—

20 gm. of crude ether asphaltenes are dissolved in 1 : 1 benzene; after standing for a week, the liquid is decanted as far as possible and filtered through a fine filter. The filtrate is concentrated to a quarter of the volume and precipitated with ether. The precipitate is filtered and washed with ether until a colourless filtrate is obtained. The ether asphaltenes are dried at 100–105° C. and brought into a U-tube, heated in a water-bath at 60° C. A stream of dry pure nitrogen is led through the asphaltenes, by which the last traces of ether and water are eliminated.

Elementary Composition of Products from Trinidad-Lake Asphalt.

	C.	H.	S.	N.	Ashes.	O (by difference).
1. Ether asphaltenes . . .	78.1	7.6	7.4	1.6	0.9	4.4
2. Difference-asphaltenes . .	78.8	8.3	5.8	1.3	0.0	5.8
3. Petroleum-spirit insoluble from high vacuum dis- tillate	74.8	6.7	4.3	2.6	0.7	10.9
4. Acetone-soluble part of the difference-asphaltenes . .	74.8	7.7	6.2	1.5	0.0	9.8

The most striking fact is the high oxygen content of the acetone-soluble part of the difference-asphaltenes. These highly oxygenated bodies are also to be found in the petroleum-spirit-insoluble part of a Trinidad-Lake asphalt high-vacuum distillate. It appears that in Trinidad-Lake asphalt the O-compounds are concentrated in the difference-asphaltenes, the S-compounds in the ether asphaltenes.

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EVALUATION OF ROTARY DRILLING LINES.*

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

INTRODUCTION.

THE main reasons for keeping records of the ton-miles done by drilling lines are :

- (1) To obtain a comparison of the service given by different brands of lines.
- (2) As a guide to the best treatment to give lines.
- (3) As an indication of how much more life may be expected from a line.

The chief objection is that it is not practical to determine the work done by certain forces such as the dynamic forces involved, or as the work done when pulling on stuck pipe. If it is considered worth while keeping records, there remains the question of what limit of accuracy is justified in view of these indeterminate stresses. In the author's opinion the determinations should be as accurate as possible, as long as they can be made quickly each day from simple charts.

A paper on the "Evaluation of Rotary-Drilling Lines" was published by K. N. Saatjian and V. V. Mason in the *Oil Weekly* of 18th November, 1935. They surveyed the effect of the various factors and equipment throughout the U.S.A., and produced an alignment chart based on a modified form of Hallam Anderson's formula. This chart has been approved by the A.P.I.

It will be seen at once that, since this chart is based on data which represent the average of all the fields in the U.S.A., it cannot be expected to give accurate results when applied to a particular field. The author therefore prepared graphs based on a modified formula to suit the conditions on the Apex field.

THE FORMULA.

The formula on which these graphs were based is as follows :

Work Done by Drill Pipe.

Let n = the number of stands.

l = length of one stand in feet.

L = length of string in feet.

w = weight of pipe in lb./ft. (with weight of tool joints prorated over the whole).

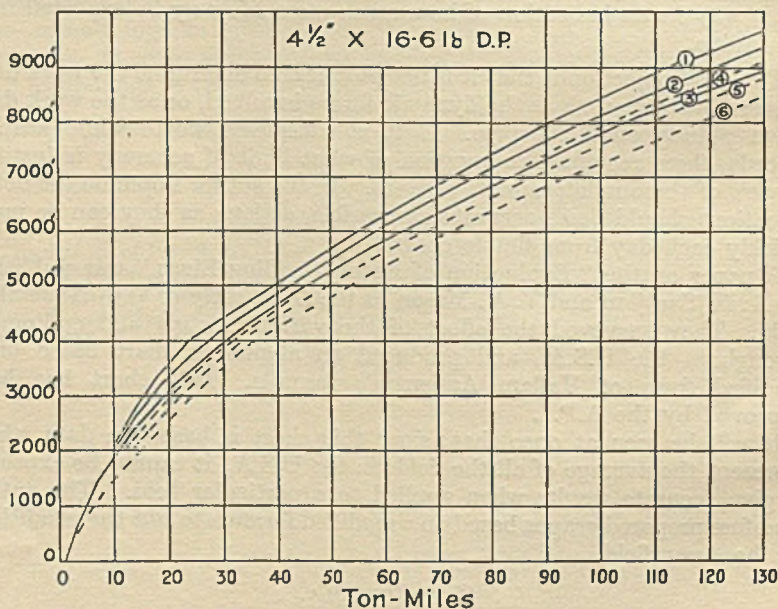
$$\begin{array}{l} \text{Running in, work done by first stand} = wl \times L \\ \text{'' '' '' '' '' 2nd ''} = wl \times (L - l) \\ \text{'' '' '' '' '' nth ''} = wl \times \{L - (n - 1)l\} \end{array}$$

* Paper read to a Meeting of the Trinidad Branch on 28th February, 1940.

By arithmetic progression, work done by whole string :

$$\begin{aligned}
 &= \frac{n}{2} [wL + w\{L - (n - 1)l\}] \\
 &= \frac{n}{2} w\{2L - (n - 1)l\} \\
 &= nwL - \frac{n^2}{2} wl^2 + \frac{n}{2} wl^2 \\
 &= wl^2 - \frac{wL^2}{2} + \frac{wLl}{2} \\
 &= \frac{wL}{2} (L + 1)
 \end{aligned}$$

APEX (TRINIDAD) OILFIELDS, LTD., TON MILE CHART.



Curve 1,	Block and hook	6,000 lb.	Mud	125 lb./cu. ft.
Curve 2,	"	" 6,000 "	" 100 "	" "
Curve 3,	"	" 6,000 "	" 75 "	" "
Curve 4,	"	" 10,000 "	" 125 "	" "
Curve 5,	"	" 10,000 "	" 100 "	" "
Curve 6,	"	" 10,000 "	" 75 "	" "

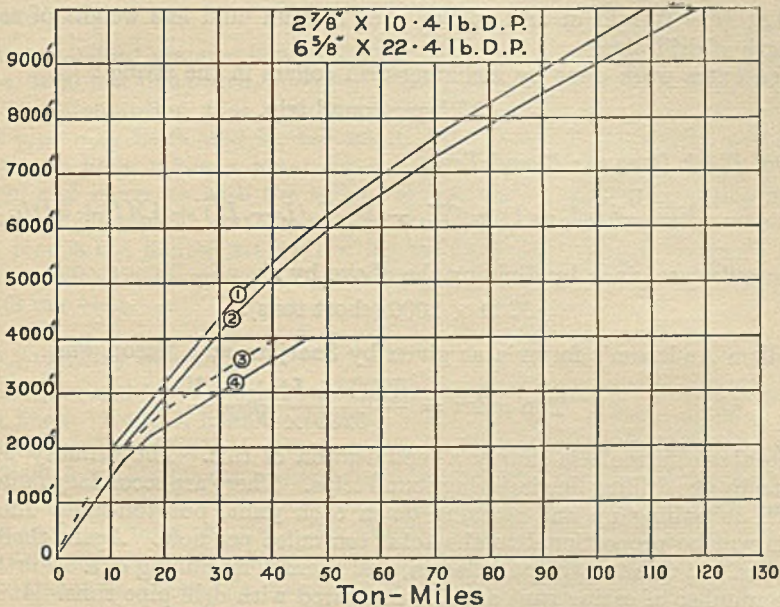
Hence work done by drill pipe for one round trip = $wL(L + 1)$.

The weight per foot must be corrected for the buoyancy of the steel in the mud. The correction is given by the volume of the steel per foot multiplied by the specific gravity of the mud. Saatjian and Mason subtracted only half this correction in their calculations. The author does not see the reason for this, and cannot agree with it. In support of this it may be stated that their tables, derived by this means, giving the weights

of drill pipe in various weights of mud, do not agree with those published in the *Petroleum Engineer's Continuous Tables* (July 1939 and subsequent issues). If the pipe is floated in, the effective weight per foot will be less again, the correction being, of course, the overall volume per foot multiplied by the specific gravity of the mud. If the pipe is floated in, but filled to the surface after a certain length has been run the formula must be altered as follows :

- Let W_1 = Weight/ft. of drill pipe in mud fluid full.
- W_0 = " " " " " " " " " " empty.
- L_1 = Depth when pipe is first filled.
- L_2 = " " " " " " " " " " filled for second time.

APEX (TRINIDAD) OILFIELDS, LTD., TON MILE CHART.



Curve 1,	2 $\frac{7}{8}$ " D.P.	Block and hook	10,000 lb.	Mud 125 lb./cu. ft.
Curve 2,	2 $\frac{7}{8}$ "	" "	" 10,000 "	" 100 " "
Curve 3,	6 $\frac{3}{8}$ "	" "	" 6,000 "	" 75 " "
Curve 4,	6 $\frac{3}{8}$ "	" "	" 10,000 "	" 75 " "

Suppose the pipe were run in all the way empty, then the work done :

$$= \frac{1}{2} W_0 L(L + 1)$$

Correction for portion filled :

$$= (W_1 - W_0)L_1(L - L_1)$$

Further correction if pipe is filled a second time :

$$= (W_1 - W)L_1(L - L_2)$$

Work done pulling out :

$$= \frac{1}{2} W_1 L(L + 1) \text{ as before.}$$

Hence for round trip :

$$\frac{W_1 + W_0}{2} L(L + 1) + (W_1 - W_0)L_1(L - L_1) + (W_1 - W_0)L_1(L - L_2).$$

Work Done by Travelling Block.

For every stand block travels	21 feet
„ „ round trip block travels	$2 - 2nl$ feet
	$= 4L$
	$= 4ML$

And work done

When M = weight of block, hook, etc.

Work Done by Drill Collars, Reamers, etc.

Let D = Weight of drill collars, etc., in mud fluid less weight of same length of drill pipe.

Then extra work done by including drill collars in the string :

$$= 2DL \text{ per round trip.}$$

Total Work Done per Round Trip.

$$= \frac{W_1 + W_0}{2} L(L + 1) + (W_1 - W_0)L_1(L - L_1) + 4ML + 2DL.$$

Ton-miles are given by dividing the above by :

$$5280 \times 2000 \text{ (short tons).}$$

Hallam Anderson's formula as given by Saatjian and Mason was :

$$\text{Ton-miles} = \frac{WL(1 + L) + 4LM}{5280 \times 2000}$$

The above formula is simply a modification of this. The primary work done by the drilling line is during round trips. There are secondary factors, such as pulling up and reaming down each joint, but ton-miles due to this will be proportional to the total ton-miles per hole. Again there is casing, but this is largely floated in, and there is no pulling out. Further, the number of casing runs are few compared with drill-pipe runs. It can therefore be seen that the number of ton-miles due to running casing will form only a small fraction of the total ton-miles. It is therefore held that by recording the ton-miles done by each round trip by means of the above formula will give a figure closely proportional to the actual ton-miles done by a wire line within the limits set at the beginning of this paper.

THE CONSTRUCTION OF GRAPHS.

For quick daily determinations a series of graphs were prepared. The many variable factors necessitate a number of curves, but for easy reading this number must be kept low. The effect of these variables was therefore investigated, and the number of curves selected for each variable was according to the magnitude of its effect. One curve was then drawn to represent the average of each set of conditions normally liable to be encountered on the Apex Field. The effect of these variables is as follows :

Weight of Mud.

Either a shale mud weighing between 72 and 78 lb./cu. ft. or a loaded mud between 90 and 110 lb./cu. ft. is normally used in this field. An average curve is therefore provided for each of these groups and one to cover the occasional use of heavy mud. The main effect of the weight of the mud is due to floating in, *e.g.*, suppose an average curve of 100 lb./cu. ft. was used instead of the 3 curves. With $4\frac{1}{2}$ -inch drill pipe at 4000 feet, a light block and 75 lb. mud, the error would be 12 per cent. if the pipe was floated in. If the pipe was not floated, the error would only be 4 per cent. Saatjian and Mason did not allow for floating in. Also they considered that effect of tool joints and buoyancy of the metal counteracted each other, and so allowed for neither.

Floating in.

The standard practice here is not to float more than 6000 feet of $2\frac{7}{8}$ -inch pipe, 4000 feet of $4\frac{1}{2}$ -inch, 2000 feet of $6\frac{3}{4}$ -inch, and the curves were drawn on the assumption that this amount was floated in. Since, however, less pipe may be floated in, but never more, it would have been perhaps better to have taken a lower figure. The effect of floating the pipe is large, and increases with the weight of the mud. Taking again the case $4\frac{1}{2}$ -inch drill pipe at 4000 feet, with a 75-lb. mud, the ton-miles done if the pipe is not floated are 34.5, if floated 29.8, or a difference of 15.7 per cent. With 125 lb. mud the respective figures are 31.9 and 24.0, a difference of 33 per cent.

The Travelling Block.

Saatjian and Mason suggest a figure of 10,000 lb. for the travelling block and hook. On this field the heavier rigs have blocks of this size but the lighter rigs have blocks weighing down to 6000 lb. With $4\frac{1}{2}$ -inch drill pipe at 2000 feet this means a difference of some 30 per cent. The effect of the travelling block at shallow depths is therefore large (with $4\frac{1}{2}$ -inch drill pipe in 75 lb. mud the ton-miles due to it are greater than those due to the drill pipe up to 3000 feet), and it must be remembered that a wire line may spend its entire life operating at shallow depths. Therefore curves were drawn for the light and heavy hooks and any intermediate values interpolated.

Drill Collars.

1500 lb. extra are allowed for drill collars with $2\frac{7}{8}$ -inch drill pipe, 2500 lb. for $4\frac{1}{2}$ -inch drill pipe and 5500 lb. with $6\frac{3}{4}$ -inch drill pipe. These weights allow for the buoyancy of the mud. At the most (say $6\frac{3}{4}$ -inch drill pipe at 2000 feet) these weights represent only 13 per cent. of the ton-miles. Hence a 50 per cent. variation in the weight of the drill collars will give only a 6 per cent. variation in ton-miles at maximum. Hence no variation in size and length of drill-collar sections was allowed for. Saatjian and Mason allow for very much heavier drill collars than these. They made a flat addition of 12 ton-miles per round trip in all determinations. This would lead to serious errors in this field, especially at shallow depths, since the total ton-miles up to 2000 feet is usually less than this.

CONCLUSIONS.

Readings made from the alignment chart of Saatjian and Mason are on a broad average, 50 per cent. higher than those obtained from the author's curves. This is quite understandable, since, as the above discussion shows, in every point of difference their methods give the higher readings.

It may be argued that one average curve for all the conditions on this field would be enough. The objection to this is that a wire line is assigned to a particular rig, and that rig often to a particular type of work, and so the line would operate all its life under one particular set of conditions. Therefore results from the single average curve would be accurate only over the average of a number of lines. A curve for each set of conditions gives a reasonably accurate figure for each line and causes little extra trouble in reading. Since the effect of the dynamic stresses must mainly depend on weight, their effect would be proportional to the static figure, and hence results should still be comparative. This, of course, presupposes that the brake is handled reasonably. If not, the line will wear much more rapidly since impulse loads are very much higher than static. For unusual strains such as pulling casing, it is suggested that a note be made against any line undergoing same.

Finally the author again emphasizes that the degree of accuracy which is justified is an open question. This paper submits one view, and it is hoped that others will express theirs. If the value of fairly accurate ton-mile records is admitted, it might be worth investigating the best method of making them comparative on an Island basis.

The author's thanks are due to Messrs. Apex (Trinidad) Oilfields, Ltd. for permission to read this paper, and to Mr. G. H. Scott and members of the Drilling Staff for information and suggestions.

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Geology and Development.

1059.* **Mississippian Border of the Eastern Interior Basin.** J. M. Woller and A. Sutton: *Bull. Amer. Ass. Petrol. Geol.*, 1940, 24, 765-858.—The Eastern Interior Basin is a sub-circular structural depression occupying much of Illinois, Indiana and Western Kentucky. Throughout the latter part of the Palæozoic era the Basin was a negative area determined by the positive Ozark Dome, Wisconsin Highland and Cincinnati Arch.

Recent classifications have divided the Mississippian system into two series: Iowa and the Chester; it is suggested that a threefold division be made: the Kinderhook, Valmeyer, and the Chester. Despite its greater thickness, the Chester probably represents a time-interval nearer to each of the proposed lower divisions.

The relations of the Mississippian and Devonian in Central U.S. are confused. Very uniform black shales separates rocks with the characteristic faunas of the two systems.

The Iowa series contains the Kinderhook, Osage and Meramec groups. In the Mississippi Valley area these are mainly cherty limestones which grade eastward into clastic sediments. The basal Kinderhook is largely shale and is generally unfossiliferous, though *Chonopectus* and *Productida* occur. The Osage group is generally unconformable on the Kinderhook. The fauna which represents a single evolutionary series includes *Syringothyris* and *Leptaena analoga*. The Warsaw and Salem limestones above are closely related and form transition beds between the Osage and Meramec groups. It is suggested that the name Warsaw be applied to beds between the Kookuk (Osage) and St. Louis (Meramec) limestones.

The Meramec contains three limestone members which give rise to large areas of karst topography. *Endothyra baileyi*, *Lithostroton canadense*, and *Platycrinus penicillus* are abundant.

The unconformity which separates the Meramec group from the Chester series is the most important stratigraphic break and is indicated by variations of the beds between the Chester beds. These consist of alternating sandstone and limestone-shale formations of variable thickness and lithology, and form three groups—the Now Design, Homburg and Elvira. The most important fossils are *Pentremites*, *Galarocrinus*, *Chonetes*, *Camarophoria* and *Eterotoerinus*.

The Chester rocks contrast with the Lower Mississippian in the wide occurrence of sands. The source of these sediments is not accurately known, though the Ozark probably contributed much to the Eastern Interior Basin. The marine beds were laid down in shallow seas and the sandstones resemble those of the Pennsylvania with thin coal-beds. A feature of the Series is the alternations of formations with form units closely resembling the Pennsylvanian "cyclothem". A marked unconformity occurs at the top of the Chester beds.

The structural history of the area indicates great Pre-Mississippian deformation. Intra-Mississippian movements gave rise to slight doming of the Ozark region.

close of the Mississippian period saw the positive areas surrounding the Basin domed and subjected to erosion.

Not only do the Mississippian rocks yield important amounts of petroleum, but they also produce much building stone. The chief oil and gas formations are listed, and the paper is accompanied by areal geological maps with a selected bibliography of more than one hundred entries. G. S. S.

1060.* Geochemical Exploration (Soil Analysis), with some Speculation about the Genesis of Oil, Gas, and other Mineral Accumulations. E. McDermott. *Bull. Amer. Ass. Petrol. Geol.*, 1940, 24, 859-881.—The rôle of surface evidences of buried accumulations of oil and gas has been much neglected. Methods of geochemical exploration permit the quantitative measurement of microscopic gas and oil seeps and mineralization phenomena which cannot be detected visually. Geochemical data show that leakage occurs from practically all buried accumulations, maximum leakage occurring over the edges of accumulations rather than over the top.

Gaseous constituents pass through microscopic fissures to the earth's surface and are subsequently adsorbed. Accompanying these, and heavier hydrocarbons, are mineral concentrations; they are the result of transportation of mineralized waters to the surface. Field results of these methods with accompanying diagrams are given.

Speculation has arisen on the origin and manner of accumulation of oil and gas. As oilfields are, with few exceptions, found in marine sediments, it is believed that salt acts as a catalytic agent in the polymerization of the migrating gases. Trask has shown that no appreciable difference exists in the organic content of marine and non-marine sediments. Experimentally, solid hydrocarbons are formed by percolating hydrocarbon gas through a salt solution.

Buried vegetable matter, basement rocks, and the earth's interior are the probable sources of the gases; these are, in ultimate origin, inorganic. The nature and amount of accumulation depend on the permeability of the cap-rock, and it appears that leakage is essentially vertical. Such cap-rocks are not necessarily source-rocks.

Very high mineral concentrations indicate the great transporting power of gases; caliche and secondary mineralization may be explained in this way. G. S. S.

1061.* Geology of the Oil-field Belt of S.W. Iran and Iraq. G. M. Lees and F. D. S. Richardson. *Geol. Mag.*, 1940, LXXVIII (3), 227-252.—The main geological features of the oil-field belt of S.W. Iran and Iraq are described. The belt includes the fields of the strongly folded Zagros foothills and those situated on the gently folded margin of the stable foreland of the Arabian littoral.

A Tertiary geosyncline extended for 1200 miles from Northern Iraq to the mouth of the Persian Gulf. Within the mountain area of the Zagros two structural zones are recognized: the normally folded zone, like the French Jura but on a larger scale, and the north-east overthrust zone. These differ lithologically, the former containing elements similar to those of the Dinarides of South-east Europe, whilst the latter are mainly metamorphics. Cambrian salt plugs are important structures in Central and South-west Iran.

Beds from the Cambrian to Recent occur, and are predominantly calcareous. The Oligocene and Lower Miocene limestone series (Asmari Limestone) is the oil-producing formation in the South-west Iranian fields, where it reaches 1500 ft. in thickness. It is overlain by the Lower Middle Miocene chemical deposits, the Lower Fars Series. Throughout the greater part of this series salt is irregularly distributed; it is known to have a thickness of 4000 ft. in certain places, and this may be exceeded elsewhere. Salt flowage occurred on a great scale during the period of the major folding movements; these resulted in the formation of various disharmonic structures, including large overfolds and overthrusts. The main north-west to south-east folding movements occurred in Upper Miocene and Pliocene times.

The oil is believed to have originated in marls deposited during Mesozoic and Palæogene times; these marls are chocolate-brown to black, evenly bedded and probably laid down in a stagnant deep-water milieu deficient in light and oxygen. Oil accumulates in the networks of the larger joints in the Limestone, the Lower Fars Series forming an effective seal. Oil or gas flows to the surface without pumping and may flow at a rate exceeding 1 million gallons a day. Middle Cretaceous limestones have also been drilled, and in the Arabian littoral these rocks contain oil.

The existing fields are situated in the foothills, especially in the wider zones in the north-west (Kirkuk) and in the south-east (Kuzistan). Seepages of oil and gas occur frequently, whilst the presence, or former presence, of gas escapes is marked by chemical changes in the surrounding rocks.

Location of accumulation is difficult because the surface structural features are often completely dissimilar to those of the Asmari limestone. This is due to the disharmonic movements of the Lower Fars, which behaved as a plastic medium between the limestone below and the relatively rigid masses of Middle and Upper Fars and Bakhtiari sediments above. It is now possible to locate the limestone anticlines by geophysical means.

G. S. S.

1062.* Sedimentation. R. M. Bailey. *Oil Wkly*, 27.5.40, 97 (12), 27.—A brief account is given of various aspects of sedimentation—the sources of the sediments and the types of weathering; the different modes of transportation of sediments by water, air, or ice; the effects of transportation on the sedimentary material; the types of deposits and their spatial relationships, and the economic applications of sedimentary studies.

G. D. H.

1063.* Photogrammetry and the Oil Engineer. H. T. Bruce. *Oil Wkly*, 10.6.40, 98 (1), 19.—The principles underlying the development of photogrammetry are briefly described, and the various instruments and their possibilities are noted. The use of photogrammetry and its advantages in pipe-line and geological work are discussed in a general manner.

G. D. H.

1064.* Michigan Alarmed Over Lag in Discoveries. N. X. Lyon. *Oil Wkly*, 17.6.40, 98 (2), 30.—Eighteen months of intensive wildcatting have yielded no important discovery, although several notable extensions have been made. Subsurface interpretation, coupled with drilling, trend geology, or chance, has accounted for virtually all Michigan's discoveries in the past. In South-west Michigan the Traverse limestone is only 1000–1900 ft. deep, so that wildcats were cheap. A table summarizes the results of wildcatting since 1931.

In 1939 697 shallow oil wells, 3 gas wells, and 463 dry holes were drilled.

The Grand Rapids field covers 3810 acres and has 386 producing Traverse lime wells. It is 6 ml. long and $\frac{1}{2}$ –2 ml. wide. Three wells produce from the Berea at 1150–1200 ft. It is the most basin-ward of all the shallow oil-fields in West and South-west Michigan. The Traverse wells are 1700–1900 ft. deep. The closure is 75–90 ft.

The Bloomingdale field produces from a depth of 1200 ft.

Since 1937 all the commercial production developed in West Michigan has been in the Traverse. Few wells have gone deeper.

The Temple pool produces from the Monroe lime at 3800–3900 ft., on the Porter-Yost–Mt. Vernon trend. A saddle divides it into two parts, and it is under hydrostatic control. The Wise field gives oil from the Dundee at 3700 ft. There has been a revival of drilling for Trenton production in South-east Michigan.

At the beginning of the year reserves were estimated at 45,000,000–55,000,000 brl., 20,000,000 brl. being in shallow pools, quantities which represent only two or three years' production at present rates.

G. D. H.

1065.* Active Development Programme Under Way in South Texas. N. Williams. *Oil Gas J.*, 13.6.40, 39 (5), 26.—Drilling has been increased in the Corpus Christi and lower Gulf Coast areas of Texas. The main activity is in the trend of fields running north from Sullivan City into Nueces county, and including the Rincon, Sun, Kelsey, and Colorado fields. At Rincon seven sands have been found. Sun, Kelsey, and Colorado are also new fields. Production has been extended at Robstown. Oil has been found at Wade City, which was formerly a gas-distillate producer at 4800–4900 ft. The Orange Grove field was opened in May. East Flour Bluff field is on the downthrown side of a fault bounding the Flour Bluff field. A gas-distillate well has been brought in at Bloomington. Vicksburg production has been discovered in Victoria county. At Telferner oil was found at 2692–2698 ft. The Keeran field has been extended, and east of Lolita another field has been opened in Jackson county.

Altogether fifteen fields have been discovered in South Texas this year.

G. D. H.

1066.* Petroleum Exploration by Means of Soil Analysis. J. W. Morrill. *Oil Gas J.*, 13.6.40, 39 (5), 68.—The history of the search for oil shows a development from drilling near seeps, through the search for anticlines to the geophysical discovery of buried structures. In recent years examination of the "soil air" seeping into shallow holes over oil-fields has shown a pattern of values marking the limits of the fields. Soil samples are now used for the same purpose. A set of high hydrocarbon values occurs over the margin of the oil body. This is the case where there is a dome, fault, shoreline, or other stratigraphic trap.

Laboratory technique seems to be advancing more rapidly than interpretation. Profiles should be sufficiently long to determine the outer margins of possible annular oil bodies. The values seem to be some indication of the productivity. Examples of the application of soil analyses are described.

Geochemistry is especially valuable in searching for shore-lines, sand lenses, and other stratigraphic traps. It seems that the geologist should find areas likely to yield stratigraphic traps, and then the geochemist can locate oil itself relatively cheaply.

G. D. H.

1067.* New Discoveries may Change Output of Illinois Fields. H. F. Simons. *Oil Gas J.*, 20.6.40, 39 (6), 17.—The Trenton limestone has been shown to be productive at Centralia at depths of about 4032 ft. After acidizing, it gave 113 bbl. of oil in 16 hr. If this declines as rapidly as the Devonian production, much better wells will be required to induce drilling to this deeper horizon.

The Trenton is productive at Dupu in Illinois, where it has yielded 1,000,000 bbl. since 1928. This field covers 670 acres, and the Trenton at a depth of about 600 ft. is highly permeable. The wells make much water, and now average only 4 bbl. of oil/day. The Westfield and Waterloo fields also give Trenton oil, and the Trenton is productive in Michigan and North-west Ohio-North-east Indiana. The latter area has had large wells.

At the present time 40% of the Illinois production is from the Devonian.

G. D. H.

1068.* Red River Counties Spurred by New Discoveries. D. Dalrymple. *Oil Gas J.*, 20.6.40, 39 (6), 20.—A few weeks ago the Cumberland pool was discovered in Oklahoma, and has led to great activity in the southern part of the State. The discovery was preceded by geological and geophysical work, and shallow tests which had oil-shows. Oil was eventually found in the Tulip Creek sands at 4952 and 5088 ft. The daily potential was 4542 bbl., and 25,000,000 cu. ft. of gas was gauged. Some 250,000 acres have now been leased in this region. The nearest pipe-line is 15 mi. away.

North-west of Madill a wildcat has found 6,000,000 cu. ft. of gas at 1120 ft. Wells are to be drilled in Atoka county, and others are under way in Cotton, Comanche, Stephens, and Tillman counties. Geophysical, geological, and geochemical work is being carried on in the Red River valley.

Oil has been known for some time in the Enos, Isom, and Kingston pools of Marshall county, and comes from depths of 400-500 ft.

G. D. H.

1069.* Drilling Activity in 1940 Approaches 1937 Peak. W. V. Howard. *Oil Gas J.*, 20.6.40, 39 (6), 25.—Completions may amount to 32,000 in U.S.A. in 1940, for total completions were 18.4% ahead of last year and drilling operations 17.8% more numerous than on June 1st, 1939. The bulk of the increase is in the eastern area, Illinois, Kansas, California, and coastal Louisiana. The drilling activity of 1939 and 1940 is compared in tables.

Arkansas, North Louisiana, and Eastern Texas show the greatest decline in activity of all the major producing areas.

G. D. H.

1070.* Steady Expansion in all Phases of Kansas Oil and Gas Operations. D. Dalrymple. *Oil Gas J.*, 27.6.40, 39 (7), 28.—Since 1860 more than a billion barrels of oil have been recovered in Kansas, and more than 400 oil and gas pools have been developed. In estimated reserves Kansas is the fifth state, and in daily average production it is sixth.

The history of the development of oil production in Kansas is described briefly. From 1905 to 1910 the annual output of oil fell from 4,250,000 bbl. to 1,128,000 bbl.,

in consequence of big discoveries in Oklahoma. Extensive developments raised production to a peak of 45,450,000 bbl. in 1918, a figure which was not surpassed until 1934. The shoestring sands were developed after 1918, and in 1922 South-west Kansas entered the picture. The Western Kansas campaign continued from 1923 to 1937, when a new high of 70,835,000 bbl. was reached under strict proration. A decline followed as a result of developments in Illinois.

A table gives the annual production since 1889.

G. D. H.

1071.* Central Kansas Uplift has Several Prolific Pays. E. A. Koester. *Oil Gas J.*, 27.6.40, 39 (7), 50.—The oil- and gas-fields of the Central Kansas Uplift are on a buried subsurface feature. It is a broadly arched feature of pro-Pennsylvanian rocks, involving pro-Cambrian. Over much of the area the Arbuckle dolomite immediately underlies the Pennsylvanian, and the porous parts of the Arbuckle serve as the principal oil horizon. The Simpson gives a little oil. The Lansing-Kansas City (Pennsylvanian) limestones have given about one-fifth of the Kansas oil and the Arbuckle more than two-thirds.

Acid treatment of Lansing-Kansas City limestone beds has met with great success. The porous zones are lenticular and erratic. Many of the best pays appear to be only 5 ft. thick, and porosities may be 30–40%. A recovery of 6000–7000 bbl./acre seems a reasonable expectation. The production is generally associated with a local high, and some of these are revealed in the surface Cretaceous beds.

Generally the best Arbuckle production is at the very top of the formation, but at times it is 20–30 ft. down, and sometimes there are several well-saturated zones. Apparently porosities of 20% are common. Acidization is beneficial, and most of the fields are under water-drive. The Arbuckle surface has some non-productive sinks. In general, oil production in the Arbuckle is associated directly with anticlinal structure. Recoveries of 7000–10,000 bbl./acre are common.

The Topeka, Howald, and Tarkio limestones of the Pennsylvanian give a little oil, and the basal sand (Gorham sand), which ordinarily lies between the Arbuckle dolomite and the pro-Cambrian granite or metamorphics, has given considerable amounts of oil in some areas. There is minor production from pro-Cambrian granite and quartzite.

G. D. H.

1072.* Kansas Oil Reserves Estimated at More Than 750,000,000 Bbl. W. V. Howard. *Oil Gas J.*, 27.6.40, 39 (7), 52.—In the first four months of 1940 the estimated proven reserves were increased by 22,484,000 bbl. A table shows the past production, estimated reserves, number of producing wells, and the producing horizons in the Kansas oil-fields. Other tables list the twenty-five fields with the largest reserves, the estimated ultimate recoveries of the twenty-five largest fields, and the total production of the twenty-five largest fields.

G. D. H.

1073.* Kansas Operations by Fields. Anon. *Oil Gas J.*, 27.6.40, 39 (7), 58.—The oil-fields of Kansas are listed, together with the producing formation and its depth, the date of discovery, production to May 1940, daily average production, and the gravity of the oil.

G. D. H.

1074.* Large Gas Reserves Ensure Many Years' Supply. J. H. Page. *Oil Gas J.*, 27.6.40, 39 (7), 69.—It appears that 15,000–25,000 gas wells, 100–2000 ft. deep, have been drilled in Eastern Kansas. Most of them are 800–1000 ft. deep. Gas occurs in sand, limestone, shale, coal, and chert, but mainly in the Bartlesville sand. A rough estimate of the reserves of Eastern Kansas gives 100 billion cu. ft.

The State's major gas reserves are, however, in the western part. Production is from the Permian lime and dolomite at Hugoton, at an average depth of 2700 ft. The original gas reserve is estimated at 13,736,375,000,000 cu. ft., of which 1.3% has been produced. At Cunningham gas is in the Viola, and the reserve is put at 80,000,000,000 cu. ft. The Medicine Lodge gas is in the Mississippi lime. At Lyons the Arbuckle lime gas production is on a buried structure revealed by seismic work. In McPherson county most of the gas is in the Mississippi lime, associated with oil.

Kansas has twenty-three natural gas pipe-line systems, with a combined mileage of 9165 ml. There are twenty-two natural gasoline plants with a capacity of 322,300 gal./day.

G. D. H.

1075.* Stratigraphy of Kansas. R. C. Moore. *Oil Gas J.*, 27.6.40, 39 (7), 73.—The pre-Cambrian does not outcrop, but it has been penetrated by wells along the Nemaha granite ridge and on the Central Kansas Uplift. It consists of granites, metamorphics, and arkose, the last containing some oil on the flanks of the Central Kansas uplift. The Cambrian to Devonian rocks are essentially conformable, the older rocks being most widespread and the younger ones often restricted to basins. In this main group are limestones, dolomites, sands, and shales. The Mississippian is mostly limestone. The black Chattanooga shale is considered by some to be a source-rock.

The Upper Carboniferous and Permian rocks are separated from the older rocks by a major unconformity, on which the older truncated rocks outcrop. The Upper Permian (Cimarron) has red beds, dolomites, and anhydrite, in distinction from the marine beds of the Lower Permian and Pennsylvanian.

Trias may occur; there is some buried Jurassic in the west, and Cretaceous rocks are distributed throughout much of Western Kansas. The Cretaceous dips generally north-west and north. There is a thin veneer of post-Cretaceous beds in places.

G. D. H.

1076.* Eastern Kansas has Produced Bulk of State's Output. D. Dalrymple. *Oil Gas J.*, 27.6.40, 39 (7), 86.—The first commercial production may have been as early as 1886, and Kansas was established as an important oil-producing State when Eldorado was discovered in 1915. The peak in drilling activity was attained in 1918, when 3750 oil and gas wells were completed. Eldorado has now given 174,635,000 bbl. of oil. Second to Eldorado in East Kansas is Augusta. Butler county has given more than 304,000,000 bbl. of oil. Both Eldorado and Augusta are anticlinal and overlie the buried Nemaha ridge. Cowley is another highly productive county, oil coming from the Shawnee, Kansas City, Bartlesville, and Arbuckle horizons. Greenwood county has provided about 130,000,000 bbl. of oil.

G. D. H.

1077.* Mississippian Limestones in Central and Eastern Kansas. W. Leo. *Oil Gas J.*, 27.6.40, 39 (7), 102.—The thickness of the Mississippian limestones in Central and Eastern Kansas bears a close relationship to the geological structure, and hence to the occurrence of oil and gas accumulations. Limestones of this age are widely distributed, but they are absent over much of the major uplifts. These beds were deposited on a nearly flat surface of Chattanooga shale, gently folded, and then eroded. The pre-Pennsylvanian folding produced pronounced anticlines. 18,000–20,000 ft. of Cherokee and earlier Pennsylvanian rocks were laid on top of the eroded Mississippian. However, some parts were not always submerged, and so porous parts were developed in the limestone and residual chert left on the surface.

Pre-Mississippian folds seem to trend north-west. The original folds of the Mississippian rocks run north and north-east. North-easterly trends predominate in the later folding. Towards the Nemaha ridge there is a tendency to *en echelon* secondary anticlines.

Nearly all the fields which produce from anticlines are underlain by thin sections of Mississippian. The producing horizons of the Mississippian seem to be independent of the stratigraphical position, and dependent solely on the porosity. G. D. H.

1078.* Forest City Basin Prospects Improved. D. Dalrymple. *Oil Gas J.*, 27.6.40, 39 (7), 107.—Oil has been found in Jefferson county in the Kansas sector of the Forest City basin. The initial well is small. Two shallow oil pools have been opened in Johnson county on the south flank of the basin, and there are six gas pools. Oil has been discovered in Richardson county, Nebraska, while there has been much leasing in Brown county, Nebraska, and also in Atchison, Doniphan, Douglas, Jackson, Jefferson, Johnson, Leavenworth, Nemaha, Osage, Pottawatomie, Shawnee, Wabaunsee, and Wyandotte counties of North-east Kansas.

West of Topeka a wildcat was abandoned at 2720 ft. in water in the Wilcox. In Johnson county oil was found in the Squirrel and Bartlesville sands in the Gardner pool and in the Bartlesville $2\frac{1}{2}$ ml. south-west of the O.K. discovery. The Arbuckle failed to give oil in tests in Douglas, Atchison, and Jefferson counties. Wildcats to the Bartlesville sand and Mississippian lime in North-east Kansas were unsuccessful. These failures were countered by a success late in 1939 near Falls City.

The position is viewed favourably, for most of the formations which produce oil

elsewhere in Kansas are present in the Forest City basin, as are many of the strata which are productive in Illinois. There are comparable structures also. Many of the deeper wells have had good showings of oil and gas, but few of the tests have been drilled on sound geological or geophysical data, or with modern equipment.

G. D. H.

1079.* One-fourth of Kansas' Area Leased for Exploration. Anon. *Oil Gas J.*, 27.6.40, 39 (7), 118.—In the first four months of 1940 it is estimated that major oil companies leased 100,000 acres, mainly on or bordering the Central Kansas uplift. Cowley county is another favoured area. There was a big leasing boom in 1928-1930, a decline, and then a revival in 1937, when about one-quarter of the State was under lease. At that date the Central Kansas uplift, the western flank of the Dodge City basin, and the Salina basin were the foremost areas of leasing.

A table gives the approximate acreage of land leased by major companies in the more active western and central counties in 1938 and 1939.

G. D. H.

1080.* Methods of Correlation in the Geology of Petroleum. O. L. Braccacini. *Bol. Inform. Petroleras*, Jan. 1940, XVII (185), 37-66.—This article discusses in detail the methods of correlation in the geology of petroleum—i.e., correlation between continents in order to determine the chronological order to which various formations belong; correlation between the stratification of various provinces in the same continent; correlation within one province, and, finally, correlation between horizons within a small area—e.g., an oil-field. Two methods are dealt with: the inorganic and the organic.

H. I. L.

1081.* Concessions Granted by Portugal. Anon. *Bol. Inform. Petroleras*, Jan. 1940, XVII (185), 35.—Particulars are given of a concession granted to the Companhia Ultramarina de Petroleos for exploitation of mineral-oil deposits in Timor Island in the region east of meridian 125° 50' E. GW. There is no restriction as regards the period during which the concession may be held, subject to the decree dated 9th December, 1909, and provided the concession is marked out within a period of five years.

H. I. L.

1082.* New Well by the Y.P.F. in Mendoza. Well Number L.3. at Lunlunta. Anon. *Bol. Inform. Petroleras*, Jan. 1940, XVII (185), 5-10.—On 22nd December, 1939, a new well was put in production, L.3., which appears to be as good as the best wells at Tupungato. Well is about 8 km. west of Barrancas, and 33 km. from Tupungato. The well gave an output of 43 m.³ at a depth of 2264 m. This result was obtained at a depth of only 13 m. drilled into the oil-bearing strata. The information is followed by a short note on the petroleum found in the Lunlunta zone.

H. I. L.

1083.* Renewal of the Periods of Five Years Granted to the Y.P.F. for the Concessions held in the Province of San Juan and in that of Salta. Anon. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 12-13.—Particulars are given of extensions to the period during which the Y.P.F. are allowed to work certain reserved areas in the two provinces above named.

H. I. L.

See also Abstract No. 1185.

Geophysics.

1084.* Remote-control Seismic Instruments. P. M. Honnell. *Oil Wkly*, 10.6.40, 98 (1), 23.—Seismic exploration of many Gulf Coast areas is seriously hampered, if not rendered totally impossible, by transportation difficulties, and remote-control seismic instruments are designed to make feasible the exploration of otherwise inaccessible regions. This is accomplished by keeping all the heavier recording equipment at a fixed location, and moving the seismometers, shot point, and transmission lines. Remote-controlled line amplifiers are used to compensate for attenuation. Two-wire lines are preferable to single wires and ground returns, on account of the elimination of ground and stray current troubles. The remote amplifiers are rugged in construction, and their construction is described. A diagram is given of the recording circuit wiring, and the use of the control panel is briefly outlined. A remote-control shooting circuit is given.

The overall electrical characteristics and performance of the remote control system approximate very closely to those of more conventional recording systems, and its flexibility is a boon in areas difficult of access.

G. D. H.

1085.* Direct Discovery of Oil Deposits Difficult. W. V. Howard. *Oil Gas J.*, 20.6.40, 39 (6), 27.—There seem to be certain difficulties in detecting at the earth's surface rays omitted by deeply buried oil, even if they exist.

Efforts have been made to locate oil by resistivity measurements, but, as electrical logging under much more favourable conditions shows, there is no certainty about such a method. The quantities to be measured are so small and the interpretation is so involved that direct discovery by gravitational methods seems impossible.

G. D. H.

Drilling.

1086.* Colloid Chemistry of Clay Drilling Fluid. A. G. Loomis, T. F. Ford, and J. F. Fidiem. *Petrol. Tech.*, May 1940, A.I.M.M.E. Tech. Pub. No. 1201, 1-12.—The outstanding problems in the further development of rotary drilling fluids are considered in the light of the colloid chemistry of clay suspensions as we know it to-day. Mechanisms are proposed for the effect of various chemicals now in use to control the rheological properties of drilling muds. It appears that viscosity-reducing chemicals are adsorbed on specific surfaces of the clay particles and that they reduce the viscosity of suspensions by destroying the portion of the absolute gel strength which is attributable to adhesion between particles. On the other hand, salts are not adsorbed, but cause gelation and coagulation because of induced changes in the interparticle ionic atmospheres.

Comparison shows that sodium pyrophosphate is one of the most effective viscosity reducers. However, amongst the polyphosphates the pyrophosphate which has the lowest molecular weight gives the least permanent effect, and as the molecular weight increases to the decaphosphate there is a corresponding increase in the permanence of the viscosity lowering. This behaviour suggests diffusion into the inner particle surfaces.

While both the elasticity and the mechanical strength of clay gels are destroyed by chemical treatment the initial gelling rate of muds in the completely de-gelled condition is not significantly changed.

On the basis of the detailed structure of clays as revealed by X-ray analysis, chemical formulae are written for colloidal clay particles and utilized to explain most of the phenomena shown by clay suspensions. The assumption of edge-to-edge contact to form structures in clay colloidal systems is apparently in accordance with experimental facts.

G. D. H.

1087.* Effects of Temperature and Pressure on Rheological Properties of Cement Slurries. R. F. Farris. *Petrol. Tech.*, May 1940, A.I.M.M.E. Tech. Pub. No. 1207, 1-14.—A thorough knowledge of the behaviour of cement slurries under elevated temperatures and pressures is necessary in order to solve properly the many problems presented in deep-well cementing operations. The high-pressure consistometer used in the tests measured the energy consumed in rotating a stirrer at a known rate.

Making comparisons in the above way, it was found that high pressure and high temperature singly or together accelerate the stiffening and setting of a cement slurry. At a pressure of 5000 lb./in.² and at 180° F., most of the cements tested stiffen too rapidly to permit proper placement in a deep well. At the same pressure and 220° F. all the cements tested stiffen beyond the limit of mobility immediately. Agitating a slurry at high pressures after rapid stiffening begins is detrimental to the strength of the set cement. The point on the consistency-time curves where rapid stiffening begins should be regarded as the maximum recommended pumping time, instead of using any assumed viscosity or consistency limit, such as 40 "poises" as a "limit of pumpability." The practical solution of many of the present and future high-pressure and high-temperature cementing problems will probably be solved when cements are improved so as to permit the placement of a uniform, mobile, and good-quality slurry at any given practical high temperature and pressure.

G. D. H.

1088.* Draw-works Shift Improves Rig-floor Conditions. E. Sterrett. *Oil Wkly*, 24.6.40, **98** (3), 21-22.—A greater derrick floor space has been obtained, with attendant decrease in hazards and increase in control of the well, by removing the draw-works entirely from the floor, setting the unit on a special foundation at ground level, and using skids firmly tied to buried anchorages through paired cables and turnbuckles.

In this revised plan of operation a cathead shaft alone is mounted above the floor level, with the vertical chain driving it enclosed on the driller's side. This mounting affords the driller close supervision of any lines being used on either end of the shaft, with consequent quicker stoppage in case of twisted or overrun line.

Other advantages are discussed.

A. H. N.

1089.* Redrilling and Workover Programme in Humble Field. N. Williams. *Oil Gas J.*, 13.6.40, **39** (5), 61.—An old lease that has been supposed to be depleted and had been practically abandoned is being reclaimed for production in a workover and redrilling programme. In less than a year production has been restored to a point that profitable return is being made to operators, thus forestalling premature abandonment of the lease and adding to the ultimate recovery.

Plans of the company attempting the project call for going back into each well, in turn, which is considered to have further potentialities, first cleaning the holes and attempting to recomplate the wells in the same sand in which they had been productive, and then, if not successful in that, testing higher sands either by perforating liner or casing at successively higher levels or by whipstocking up the hole and sidetracking with a new hole to a prospective sand.

The paper is a record of the operations carried out.

A. H. N.

1090.* Unitized Equipment Cuts Cost of Moving. D. H. Stormont. *Oil Gas J.*, 20.6.40, **39** (6), 46.—Through assembly of drilling equipment into highly unitized sections, each mounted on skids, and arranging the various units so that they may be set up or torn down with a minimum of time and effort, a drilling company has effected a substantial saving in the time required for moving one of its rotary rigs. The various sections are trucked from location to location without dismantling, and the derrick is usually left intact and skidded. This not only permits a saving in time and costs, but also acts to prolong the life of some equipment that sometimes is damaged each time the rig is dismantled.

In addition to the unitization features contained in the rig, effort was made towards incorporating certain safety features in the design, such as a device for automatically throwing the draw-works out of gear if the cathead rope starts piling up on the cathead.

The rig is fully described, and dimensions of major items are given.

A. H. N.

1091.* Rotary and Cable Tools used in Kansas Drilling. H. F. Simons. *Oil Gas J.*, 27.6.40, **39** (7), 48.—Equal opportunity exists in Kansas for both rotary and cable tools, although the former drills most of the hole, whilst the latter is mainly used for completion. Speed of drilling gives the rotary rig a decided advantage in drilling to the pay zone, and it is for this reason that rotaries are used where there is competitive development. It takes approximately 15 days to rig up a rotary, drill the hole, and set the pay string on the average well of 3300 ft.

There have, however, been numerous advances in both methods in recent years. Portability and small holes are two of the most important problems at present. The trouble in cable-tool drilling is mainly occasioned by the caving formations, a black and blue shale at 2100-2400 ft. giving the greatest trouble. Caving is prevented to a certain extent by the addition of salt to the drilling water.

A. H. N.

1092.* Economics of Slim-Hole Drilling. F. W. Heiser. *Oil Gas J.*, 27.6.40, **39** (7), 56.—Slim-hole drilling, having no other feature superior to methods used in the past, is warranted purely from a financial point of view. The total saving being effected in Kansas by the use of slim-hole drilling and lighter pumping equipment amounts to \$12,000 over the conventional type wells using heavy-duty equipment, as was the practice two years ago.

The advent of slim-hole drilling into Kansas was in answer to a wide demand for

reduction in development costs that would make possible development of acreage which would not be commercially attractive with the usual development costs.

Despite the acute proration situation, Kansas drilling activity again shows a progressive increase; probably due, in part, to the successful application of slim-hole drilling. The introduction of lighter pumping equipment in slim-holes has not only reduced the initial cost to a minimum, but is also reducing operating costs.

A. H. N.

1093.* Rate of Penetration in Drilling. K. C. Slater. *Petrol. Engr*, June 1940, 11 (9), 47-48.—A general discussion is made of some of the factors recognized as important in their effect on the rate of penetration in rotary drilling. A study of well records shows that during the six-year period 1932-1938 the total increase in rate of penetration in a group of more than 500 rotary wells in different fields was 123.7%. The respective percentages contributing to the total increase in rate of penetration in this group of wells was reported as follows: personnel and equipment, 55.5%; rotating speed, 39.6%; and drilling fluid, volume, 28.6%. In these figures was included another factor, weight on bit, which was found difficult to evaluate separately. The percentages shown are merely intended to suggest the importance of the parts played by the several factors in increasing the rate of penetration in the group of more than 500 wells studied. It is presumed that in different localities the figures may be different, as the character of the formations being drilled is an important factor. (Cf. J. E. Brantly and E. H. Clayton, *A.P.I. Drilling and Production Practice*, 1939.)

A. H. N.

1094.* Deepening and Completing a Well in the Lisbon Field, Louisiana. Part 5. P. D. Torrey and F. H. Miller. *Petrol. Engr*, June 1940, 11 (9), 68.—In this exhaustive paper the descriptions given in the first four parts about every step undertaken in deepening and completing a certain well are continued with the same attention to every detail. The completion programme is continued by giving the testing of the remaining sands. Finally a summary of the completion programme is given with hints for similar operations in the future.

This part concludes with data and procedure for production tests. These tests give daily gas production during test and separated liquid characteristics in terms of accumulation, recovery and shrinkage, and solubility of gas. Graphs are reproduced and discussed.

A. H. N.

1095. Modern Drilling. W. C. Bailey. *California Oilfields*, Vol. 23, No. 4, April 1940.—The stated purpose of this article is to assist operators desirous of improving their drilling technique by explaining certain new ideas and mechanical advances made in methods of drilling during recent years.

Mention is made of a direct-acting hand pump designed for use where it is desirable to avoid steam pressures exceeding 250 lb. and yet to be in a position to deliver the desired quantity of fluid against the pressures encountered.

There is a new superheated steam generator, entirely self-contained, and mounted on steel skids for portability, which is an embodiment of the principle that increased output is obtainable by more effective use of heating surface and does not necessarily involve increased size and weight.

As regards general equipment, modern designers are agreed that this should incorporate as few units as moving facilities will permit. There is one highly unitized system designed to drill to 3000 ft. which utilizes two vertical two-cylinder variable cut-off steam engines for power. While drilling, one engine only is used to drive the rotary table and the other to drive the hand pump. One boiler is used to generate the steam.

The hydromatic brake is an example of new ideas which in a very short time have become almost standard equipment. It does not obviate the necessity of mechanical brakes, but used in conjunction with them it has decided advantages and affords an extra safeguard when handling great loads.

Power is generated in Californian oilfields most commonly by steam engines. There is, however, a tendency to employ the ordinary horizontal type of engine, while actually there is available a much more efficient type involving the use of a variable cut-off arrangement instead of a throttle valve for reducing power output. For fairly deep

drilling the internal-combustion engine utilizing gas, gasoline, or diesel oil is frequently employed. Hitherto it has been regarded as inferior to steam-powered outfits, largely owing to the unsatisfactory performance of any available clutch and reverse mechanism. Correction of this fault has, however, entitled this type of engine to be favourably compared with steam-operated units.

Another invention which has effected an enormous saving in time and money is the formation tester, which obviates the laborious practice of setting a string of casing whenever a formation has to be tested. This machine in effect utilizes the drill pipe as a temporary casing, and there are two types available: one depending on mechanical means to expand a packer against the walls of the hole, and the other depending on a previously prepared seat in the formations immediately above those being tested.

Potentialities of the Turbo drill, a new and completely different method of rotary drilling, the use of gravel packing and the employment of a column of dead oil instead of mud fluid are also discussed, among a variety of other subjects connected with modern drilling practice.

H. B. M.

1096. Drilling in with Oil in Kettleman Hills Oilfield. F. C. Hodges. *California Oilfields*, Vol. 23, No. 4, April 1940.—Drilling mud, although almost invariably used in rotary drilling, has certain serious disadvantages. In partly depleted oil zones where pressures are comparatively low it tends to preclude complete recovery by clogging the interspaces in the oil sands: where pressures are exceptionally low it has been known entirely to prevent the entrance of oil or gas into the well.

Recently attention has been turned to the possibility of using oil as an alternative to drilling mud, and in this paper an account is given of routine experiments conducted by the Kettleman North Dome Association and the Standard Oil Co. of California in the Kettleman Hills oilfield.

Several major problems were encountered during the course of these experiments. In order to remove bit cuttings the stream velocity of the fluid column had to be increased. This could only be achieved by the injection of gas into the oil stream. Again, an oil-gas mixture, unlike rotary mud, is entirely lacking in wall-building properties, and the conditioning of the walls of the borehole presented considerable difficulty when this fluid was used.

Experiments with drilling equipment in accordance with conventional rotary tables proved unsatisfactory, and special equipment accordingly was more or less standardized.

The special equipment is installed after the water string is cemented and before the cement is drilled out. When the cement is drilled out and the water shut off is approved, the rotary mud is circulated out of the hole with water and the water replaced with 33–35° gravity oil. The gas is injected into the oil stream after the fluid leaves the pumps at a rate of approximately 200,000 cub. ft. per day. The North Dome Association employs a pressure of 1200 lb. per square inch and the Standard Oil Co. 900 lb. per square inch.

The sole reason for using oil instead of drilling mud is to secure better well completions, and thus increase production. Owing to the small number of wells drilled in with oil and the relatively short time they have been on production, it is not possible accurately to assess such increases. Nevertheless, conclusions based on comparison of maximum production rates attained during the early lives of these wells indicate that increased productivity is obtained from wells completed in this way. Tabulation of data concerning seven wells completed by using oil and injected gas, and wells using rotary mud completed at about the same time, in similar structural positions and in the same locality and zone, shows that six of these had a greater production than those drilled with mud. Three of them definitely had no advantages in pressure or producing conditions, indicating that the increased maximum production rates could be attributed to the method of completion.

H. B. M.

1097. Drilling. C. R. Dalo. U.S.P. 2,203,720, 11.6.40. Appl. 10.12.34. Apparatus for detecting water intrusion into boreholes utilizing a photo-electric cell and a microammeter.

J. J. Jakosky and P. B. Lyons. U.S.P. 2,203,729, 11.6.40. Appl. 19.11.38.

Method and apparatus for use in determining the geologic nature and characteristics of a formation traversed by a borehole.

C. H. Johnson. U.S.P. 2,203,730, 11.6.40. Appl. 27.1.39. Method and apparatus for determining the strike and dip of subsurface strata traversed by a borehole.

H. D. Sandstone. U.S.P. 2,203,747, 11.6.40. Appl. 20.9.37. Laminated disc drill bit.

T. H. Staneliff. U.S.P. 2,203,846, 11.6.40. Appl. 13.12.38. Drill bit with side cutters.

G. A. Lowroy. U.S.P. 2,204,091, 11.6.40. Appl. 13.2.39. Inside pipe-cutter, adapted to cut by rotation pipes inside a well.

B. Engstrom. U.S.P. 2,204,130, 11.6.40. Appl. 15.9.38. Pipe-thread protector.

A. G. H. Straatman. U.S.P. 2,204,196, 11.6.40. Appl. 7.11.38. Method and apparatus for determining the nature of borehole fluids which are substantially electrolytic, using a high-frequency oscillatory circuit.

J. R. Bradshaw. U.S.P. 2,204,340, 11.6.40. Appl. 1.6.39. Releasable floating valve for use in pipe in oil wells.

R. E. O'Donnoll. U.S.P. 2,204,378, 11.6.40. Appl. 8.6.39. Open-hole packer for temporarily securing and sealing a conduit in a well.

H. P. Wickersham. U.S.P. 2,204,458, 11.6.40. Appl. 20.3.31. Jarring tool for use in a well string.

H. E. Grau. U.S.P. 2,204,586, 18.6.40. Appl. 15.6.38. Safety tool joint.

L. F. Baash. U.S.P. 2,204,545, 18.6.40. Appl. 17.12.38. Rotary-table assembly.

L. F. Baash. U.S.P.P. 2,204,646 and 2,204,647, 18.6.40. Appl. 17.12.38 (both). Rotary-table bushings.

R. C. Baker. U.S.P. 2,204,648, 18.6.40. Appl. 5.5.39. Packing for well devices, used in particular with a cement retainer.

C. Brendel. U.S.P. 2,204,657, 18.6.40. Appl. 12.7.38. Roller bit.

C. E. Burt. U.S.P. 2,204,658, 18.6.40. Appl. 12.12.38. Well-cementing device.

C. E. Burt. U.S.P. 2,204,659, 18.6.40. Appl. 23.12.39. Slip for oil-well tools.

C. White. U.S.P. 2,204,713, 18.6.40. Appl. 20.5.39. Portable derrick fixed on a mobile base, attached to a lorry.

W. B. Woody. U.S.P. 2,204,716, 18.6.40. Appl. 5.5.38. Portable derrick mast.

W. M. Frame. U.S.P. 2,204,754, 18.6.40. Appl. 29.12.38. Threaded joint for thin-walled oil-well casing.

J. F. Chappoll. U.S.P. 2,204,844, 18.6.40. Appl. 5.12.38. Drilling and coring apparatus having a reciprocating inner bit within a rotating drill pipe.

G. F. Lo Bus. U.S.P. 2,204,938, 18.6.40. Appl. 4.5.38. Cable and rope winding device.

G. Tipter. U.S.P. 2,205,022, 18.6.40. Appl. 27.12.37. Bore-hole direction tester using heavy pendulum, a record sheet and a magnetic compass.

E. B. Hall and A. L. Armentrout. U.S.P. 2,205,119, 18.6.40. Appl. 17.4.39. Method of setting drillable liner in wells, using cement.

J. H. Burt. U.S.P. 2,205,238, 18.6.40. Appl. 19.6.39. Cable-tool bit.

J. S. Morgan and J. D. Spalding. U.S.P. 2,205,269, 18.6.40. Appl. 1.3.37. Rotary machine.

C. C. Scharpenberg. U.S.P. 2,205,697, 25.6.40. Appl. 6.5.38. Tool joint for well drilling fitted with groove of reduced thickness so that the sleeve is as flexible as the pipes it couples.

D. B. Monroe. U.S.P. 2,205,729, 25.6.40. Appl. 1.12.38. Inclinator utilizing a plumb bob, A. H. N.

Production.

1098.* **Steady Flow of Gas-Oil-Water Mixtures through Unconsolidated Sands.** M. C. Leverett and W. B. Lewis. *Petrol. Tech.*, May 1940, A.I.M.M.E. Tech. Pub. No. 1206, 1-9.—The apparatus used was similar to that employed by Leverett in investigating the flow of oil-water mixtures through unconsolidated sands. Facilities were provided for determining the oil, gas, and water contents of the sands, as well as the rates of flow. It was found that the relative permeability to water was fixed by the water saturation alone and was not affected by the introduction of an additional non-aqueous phase. The relative permeability to gas in three-phase flow is slightly less than would correspond to the same gas saturation in two-phase flow, but the relative permeability to oil varies in a more complex manner, being in some regions less and in others more than for the same oil saturations in two-phase flow. The isoperms for all components are independent of the viscosity of the oil phase. The presence of appreciable amounts of all three phases in a flowing stream in equilibrium with the fluid in the pore space is limited to a relatively small region of pore composition.

It appears that the presence or absence of connate water is not an important factor in the recovery of oil by the gas expansion mechanism. This was confirmed by direct experiment. The unrecovered oil will vary inversely as the connate water saturation.

The mechanism of three-phase flow is discussed.

G. D. H.

1099.* **Handling of High Pressures at La Gloria, Texas, Field.** H. F. Harris. *Oil Gas J.*, 13.6.40, 39 (5), 58-59.—Encountering high gas pressures results in one of the main types of difficulties in drilling deep wells on the Gulf Coast, and the same pressures often cause considerable trouble when a well is completed and attempts are made to produce it. This has led in some cases to unusual well installations, well heads, and separators in an effort to obtain production from the well both economically and efficiently. The paper describes the arrangements of chokes and heaters for expanding the gas, braden-head installations, high- and low-pressure separators, and recording orifice-type meters. Data of pressure readings are given showing shut-in pressures on the wells from 2490 up to 3650 lb./sq. in., tubing pressures while flowing ranging from 1500 to 2000 lb./sq. in., etc.

The paper is descriptive.

A. H. N.

1100.* **Metals in Production.** W. L. Nelson. *Oil Gas J.*, 13.6.40, 39 (5), 70.—This is the sixteenth of a series of articles pertaining to the use of metals and alloys in the petroleum industry, and deals with pull rods, meter parts and instruments, pumping jacks, speed-reducer gears and parts, sling or construction chain, field tanks, and water-disposal lines. The percentage compositions in terms of carbon, nickel, chromium, molybdenum, copper, and "others," as well as the yield point, elongation in 2 in., and Brinell hardness, are given for various metals and alloys used in these parts. The materials include carbon and manganese steels, Monel metal, nickel silver, bronze, brass, invar, etc.

Notes are also given regarding the use and care of these metals in their respective rôles.

A. H. N.

1101.* **Wider Spacing Places Kansas on more Profitable Basis.** H. F. Simons. *Oil Gas J.*, 20.6.40, 39 (6), 34.—Granting of full consideration to acreage up to 20 acres in

the allocation of production has permitted Kansas operators to increase their daily revenue and total income per well without an increase in investment.

A short description is given of Kansas fields, their mode of development and spacings. Costs are given for lease acreage and for drilling and developing activities; a good average figure for well investment appears to be \$22,500, to which \$4500 must be added for production equipment. Other financial and economical data are given in evaluating the profits and loss of wells in those fields.

To improve the balance the operator must increase the per-well income and/or decrease the costs. Of the three methods of increasing the income per well, two, a higher price/brl. and a greater market, are beyond the powers of the operators. Wider spacing will increase the per-well allowable providing acreage is given consideration in allocating the production.

Costs of wells have been decreased in Kansas by drilling of smaller holes, use of smaller casing and surface equipment, and wider spacing of wells. The latter results in more oil/day and more total recovery/well.

The paper is an explanation of these statements in terms of factual records of results already achieved and by a study of the various fields adopting these principles.

A. H. N.

1102.* Recognizing and Overcoming Gas Lock in Pumping Wells. T. P. Sanders. *Oil Gas J.*, 20.6.40, 39 (6), 43.—Gas lock can be recognized by low pumping efficiency, provided that enough is known of the particular well to eliminate other possible causes for poor recovery, such as insufficient pump submergence, worn working parts, leaking pump-valves or tubing, or loss of plunger stroke due to rod stretch or tubing movement.

Nine methods are outlined for overcoming gas lock: including the use of a "gas valve" at the bottom of the plunger; correcting the pump spacing; using an oversized or stream-lined standing valve; slowing down the pump; using smaller plunger and pump than the existing ones; using a gas anchor; maintaining minimum pressure on the casing head; allowing sufficient space around the pump and tubing to permit free escape of gas; and drilling a pocket or sump hole into the barren rock below the oil horizon.

A. H. N.

1103.* Draw-down Method of Taking Potentials in Kansas. M. Brown. *Oil Gas J.*, 27.6.40, 39 (7), 63.—The theory and practice of draw-down method of taking potentials is discussed. It is concluded that this method of measurement is not only a yardstick of well comparison, but also is a means of studying the action of the reservoir and the underground equipment. So impressed are certain major operators that they are purchasing the instruments not for state potential determinations, but for their own private periodic investigation of their wells' performances.

Production practice has been greatly improved by the adoption of the draw-down method of taking potentials. In addition to the scientific information which it makes available, economic and reservoir waste is reduced to a minimum.

Careful study of the collective well records on any lease is essential to find the best, and often least expensive, equipment that will satisfactorily handle the specific production problem in hand. Changes in rates of withdrawal in daily operation can often be made which will show economy in amounts of water handled, or the records may indicate necessary tubing changes in their depths or size, or recommend more economical gas-oil ratios to be obtained.

A. H. N.

1104.* Water-Flooding in Kansas. P. Reed. *Oil Gas J.*, 27.6.40, 39 (7), 77-78.—The history and operation of the main water-flooding projects in the State of Kansas are outlined. Water is pumped from a river, in most cases, by centrifugal pumps and treated with sodium aluminate, lime, copper sulphate, and chlorine. After passing through filters, the water is pumped to intake wells. Injection pressures vary from 300 to 750 lb./sq. in. Successful flooding is conducted in this area with the injection of 10 brl. of water/1 brl. of oil. Wells are drilled on a five-spot pattern with spacing of 440 ft. (north and south) and 330 ft. (east and west). Coring is important in furnishing data for the development programmes. A few wells are flowed, but many wells are pumped.

A. H. N.

1105.* Most of Kansas' Output Comes from Water-Drive Fields. E. A. Stephenson. *Oil Gas J.*, 27.6.40, **39** (7), 79.—While many of the problems of production engineering are not encountered in Kansas, such as heaving shales and extremely high formation pressure, together with the completion and operating problems which accompany these pressures, a number of developments have become almost characteristic of Kansas. The more important of these characteristics are: (1) slim-hole drilling; (2) increased portability, compactness, and unitization of machinery for drilling purposes; (3) trend towards other production methods than by the use of ordinary reciprocating pumps; (4) increasing recognition of the value of edge- and bottom-water as agents in oil recovery; (5) general acceptance of the possibility that in water-drive pools wide spacing, within certain limits, is not only economically sound, but may also result in as large per-acre recoveries as can be obtained from the long-established "rule of thumb" 10-acre plan; (6) widespread use of draw-down methods, sonic reflections, for the measurement of potential; and (7) increased use of wells drilled for special purpose of salt-water disposal.

A. H. N.

1106.* Kansas Conservation. T. A. Morgan. *Oil Gas J.*, 27.6.40, **39** (7), 113.—The effects of proration measures on the oil industry of the State of Kansas are discussed in a general manner.

A. H. N.

1107.* Kansas Wells Benefited by Slower Withdrawals. H. F. Simons. *Oil Gas J.*, 27.6.40, **39** (7), 114.—Faced with a declining market for the past 3 years and a steady diminution of the daily average production, operators in Kansas have had to rely on a reduction in the operating costs to keep their businesses profitable. Practically all of the producing practices in the area are dictated by that necessity, including reduction in size of casing, tubing, surface equipment, acidizing costs, oil treatment and water disposal, and the prevention of corrosion and unnecessary water encroachment. These items are discussed.

Since the method of taking potentials was changed, the necessity of large pumping equipment has been eliminated and it has been found that the wells equipped with small units and operated over a longer period per day have had less water trouble. The method has not only halted water encroachment, but actually reduced the quantity of water and increased that of oil produced from a well.

A. H. N.

1108.* Proper Selection of Prime Mover Leads to Pumping Economy. T. P. Sanders. *Oil Gas J.*, 27.6.40, **39** (7), 124.—The assumption that the horse-power of a prime mover should correspond to the nominal horse-power-rating of the pumping unit may lead to a poor selection. Actually the size of prime mover needed should be determined directly from the horse-power required at the polished rod, allowing a suitable factor for overall efficiency of the pumping unit. Ageing of the field results in increased average polished-rod load. It is therefore recommended that a prime mover should be installed capable of pumping fluid of the weight of water from the total depth of the well at the desired rate with no assistance offered by fluid level.

A formula, based on the study of a great many dynamometer cards—i.e., polished-rod horse-power—is given to calculate the required horse-power to pump fluids at certain rates. Surface equipment efficiency appears to be 85%. Other items, such as crookedness of holes, require more power.

A. H. N.

1109.* Optimum Spacing of Dolomite Acreage in the Texas Panhandle. P. J. Jones. *Oil Wkly*, 10.6.40, **98** (1), 28.—Paper presented before American Petroleum Institute. The author presents data showing that ultimate recovery within 10- to 20-acre limits on the Panhandle dolomite is independent of spacing. The basis for calculation is the following set of representative physical data: an allowable of 20 brls./day/well, irrespective of spacing; 365 operating days/year; an economic limit of 3 brls./day; an investment of \$22,500/well; an operating cost of \$1800/well/year, including taxes; a one-eighth royalty; and interest at 6%.

It appears that, on an average, optimum spacing of the dolomite Panhandle acreage is 18 acres/well at the present income of \$0.98/brl. of oil produced. As the recovery available from eighteen average acres is 136,000 brls., and as per acre recoveries vary,

the more general conclusion reads thus: Optimum spacing on the dolomite trend is 1 well/136,000 brls. of recoverable oil. This conclusion does not hold if the allowable is not on a per well basis. A. H. N.

1110.* "Sand Squeezing" by Air Pressure. F. R. Cozzens. *Oil Wkly*, 10.6.40, 98 (1), 42.—"Sand squeezing" is a process used to reclaim oil reserves which have been pushed back into the sand by former pressuring. Porous sands are broken occasionally by harder streaks, and it is reasonable to assume in certain cases that air under pressure in a repressuring project has pushed the fluid in porous channels and dammed it up against the hard barriers.

"Sand squeezing" consists, then, of pumping air under pressure through two wells near the hard streak, and thus forcing the oil back to the central porous streak, where wells are pumped vigorously to lift the oil thus reclaimed. The pressure of the air in each of the intake wells should be as nearly equal to the other as possible. By this method thousands of brls. of oil are often reclaimed from stretches of sand less than a mile in length. A. H. N.

1111.* Paraffin Removal Economically by Electrical Process. B. Mills. *Oil Wkly*, 17.6.40, 98 (2), 15-17.—A new method for removing paraffin deposits, by heating the tubing electrically, has been used with sufficient success to warrant installation of permanent units on thirty pumping wells over a 2-year period.

The tubing hanger is insulated from the casing-head by an insulating medium shaped to fit the heads, whilst the tubing is insulated from the casing by conical-shaped insulators placed around the couplings to hold the tubing in spaced relationship to the casing. A spring contact is set at the required depth to complete the circuit between the tubing and casing. A d.c. or a.c. of high amperage and comparatively low voltage is introduced by connecting the input terminal to the tubing and the return to the casing. Heat is generated in the tubing, rather than in the casing, because of the smaller diameter of the tubing and its consequent greater resistance to the current.

The unit is described and illustrated in detail.

A. H. N.

1112.* To Try Pressure Maintenance on Small, Shallow Tract. F. B. Taylor. *Oil Wkly*, 17.6.40, 98 (2), 18-20.—Of the more than 200 pressure-injection operations in the upper Mid-Continent area, only a small number are pressure-maintenance type of projects, by far the majority being some type of repressuring work. Of these few one, the most recent to get under way, departs from the ordinary in a number of respects. Foremost in interest is the apparent small quantity of productive acreage involved. Moreover, the project is not expected to increase profit until a more advanced stage is reached. As the project at the present time is only in its initiative stage, the paper analyses only the primary elements towards getting the work under way. However, it is already established that the decision to maintain reservoir pressure in order to increase the ultimate recovery appears to be well founded. A. H. N.

1113.* Waste in Oil Reservoirs. S. F. Shaw. *Oil Wkly*, 17.6.40, 98 (2), 21.—In this long paper the author finds it difficult to define exactly oilfield waste. There are losses of oil which are unavoidable; oil remaining currently unrecovered in the reservoir that will be recovered at some later time. It is hard to decide how much of this is "absolute waste," a term applied to such losses as oil running down creeks and not recovered, oil burned in wells that catch fire, etc.

The reasons why early primary-recovery percentages in relatively shallow fields were low are detailed, and it is also shown that in reality the recoveries were underestimated. However, it is not correct to assume that oil is wasted when it is allowed to remain in the reservoir because it is unprofitable at a given time to extract it. Secondary extraction methods have increased the ultimate recovery in certain fields by a considerable percentage. On the other hand, secondary recovery methods are sometimes shown to be unprofitable due, perhaps, in most cases to the fact that the price received for the oil is lower than has been expected. Thus higher ultimate recovery is not the only criterion that controls the action of the operator.

It is pointed out that for the last 15 years the kind of waste as oil overflowing from tanks, escape from leaky pipe, evaporation from stock tanks, burning of oil unacceptable to pipe-line companies, blowing out of wells and consequent escape of oil and gas in immense quantities, improper handling of wells, etc., has been practically eliminated. The author asserts that since 1926 he has not seen any appreciable evidence of avoidable waste in the Americas or Europe, despite statements in the Press to the contrary. Where wild wells blew out, the owners were more anxious than anybody else to put them under control.

Increase in ultimate recovery brought about by natural water drive, by restriction of production, by attempts to change gas-oil ratios without regard to the conditions in the reservoir, is of doubtful benefit. Cases are cited to illustrate the statement. In certain fields, where the formation is very porous and permeable, and where production at maximum capacity does not increase the gas-oil ratios, it is believed that ultimate recoveries are raised if the fields are produced at maximum capacities. Cases are cited to explain this and other statements in detail. A. H. N.

1114.* Skid-Mounted Pump Units Move from Well to Well. Anon. *Oil Wkly*, 17.6.40, 98 (2), 44.—Details are given of skid-mounted light-weight pumping units. In considering the economics of the practice, a number of units that have been in steady service for some months have shown that the wear is not unduly high. However, the loads must be watched and the machine must not be over-taxed. A. H. N.

1115.* Water Flood Restores Profit to Stripper Operations. J. C. Albright. *Oil Wkly*, 24.6.40, 98 (3), 15-20.—Records are now available to show that a scientifically conducted water flood will recover more oil within 4 or 5 years than has been possible to produce from many sands during their entire previous lives. A typical field is studied in some detail, describing the wells, the surface fittings, sources of water supply, and methods of its injection and measurement, tanks, power, units etc.

The quantity of oil which may be recovered from a given sand obviously depends on the quantity of residual oil in the sand at the time the flood was begun. Methods in use prior to a water flood usually depleted the sand of its gas and attendant lifting power, and may recover as much as 30-35% of the amount of oil originally in the sand when the well was first drilled. The percentage of oil recovered from the residual quantity in the sand at the beginning of water-flooding may also vary, but when a flood has been started, the wall of water should be continued at a constant pressure and input rate. Otherwise, the water may flatten out in the vertical section, and when started again could cause excessive trapping so that oil may never be brought to the surface. A. H. N.

1116.* Effects of Rod Vibration on Dynamometer Card. K. N. Mills. *Oil Wkly*, 24.6.40, 98 (3), 23.—It was noticed for a considerable time that dynamometer cards obtained for sucker rods working at low speeds were very similar to the ideal card in general. On increasing the pumping speed, from 6 to 11 strokes/min., the shape was distorted to a great extent, due to vibrations being set up in the rods.

The system may be represented by a long rod, fixed at one end, and the other end of which is subjected to suddenly applied force. By eliminating the cases of higher frequencies—i.e., assuming the damping effect of the oil to allow only the lowest possible frequency to operate—the equation of frequency of vibration is reduced simply to $f = \frac{V}{4l}$, where f = frequency, in cycles/sec., V = velocity of sound in the material of the rod string, in ft./sec., and l = length of rod string in feet. Practical tests prove this to be very nearly true. Summations of vibrations of various amplitudes and in or out of phase with each other are discussed generally.

All vibrations in the rod strings emanate from the opening and closing of the traveling valve. Practical proof is given. The rod string in a pump pumping gaseous fluids will have a vibration set up in it once every cycle, at the time the load is taken up by the string—i.e., when standing pump opens. When the same string is used in pumping a dead fluid there will be two vibrations, one when the load is taken up by the string and one when the load is taken off it. The amplitude in the case of pumping dead fluids will, therefore, be greater than when pumping gaseous fluids.

The vibrations as recorded by a surface card will be approximately half a period out of phase with those recorded at the pump due to time lag of forces applied to the ends of a string. The time of the upstroke as compared with the downstroke will further complicate the diagram.

Finally, resonance conditions are studied with the aid of diagrams built from simple assumptions to yield different types of dynamometer cards showing one or more waves either on the upstroke or downstroke portions of the card only or on both. For simplicity, only boundary conditions are studied, but these may be used as guides for more complex ones.

A. H. N.

1117.* Sand Pack Solves Well Sanding Problem. C. Johnston. *Oil Wkly*, 24.6.40, 98 (3), 34.—A well, drilled on a salt-dome flank and completed in an unidentified sand, gave severe trouble with very fine sand which, it appeared, was almost impossible to exclude. After setting ten unsuccessful screens, an eleventh and new type one was tried and found successful. This consisted of inner and outer shells of specially fabricated screen pipes containing carefully proportioned packing of sand in the annulus. Details are presented. The well is still producing, in June 1940, the same amount of fluid as it flowed when originally equipped with the screen on 14th October, 1939.

A. H. N.

1118.* Permian Basin Present Situation and Outlook. H. C. Wiess. *Oil Wkly*, 1.7.40, 98 (4), 14-16.—In discussing current problems confronting West Texas operators, the author also presents an interesting review of the situation existing in many other parts of the U.S.A. It is asserted that continuation of an active drilling campaign during a time when market demand has failed to gain appreciably has resulted in a steady decline in per well allowables until a much longer investment payout rate has been created.

Operators are urged to consider carefully the rate and circumstances under which additional drilling investments should be made, and the necessity is advocated for wider well spacing and careful development generally in order to insure profitable operations. The alternative of sound production engineering resulting in a long period of profits is a short period of boom prosperity followed by complete collapse.

A. H. N.

1119.* Charts Simplify Many Pumping Problems. B. Altman. *Oil Wkly*, 1.7.40, 98 (4), 17-23.—The principles involved in specifying the capacity, horse-power, and other characteristics of an oil-well pumping unit required to lift a certain quantity of oil per day are outlined. A simple chart gives the first case of pumping problem where ideal conditions only are assumed.

A second and more accurate chart is reproduced by means of which the number of strokes per minute and length of stroke may be determined for most economical lifting of a quantity of oil with a pump of any efficiency between 60 and 100%. Further, six charts are given for calculating the combined rod movement and tubing stretch for various sizes of rod and tubing combinations. Another graph is designed for determining the static weight of wells for any set of constants of plunger diameter, depth of well, rod diameter and oil gravity. Tables give impulse factors, and a final chart is unitized for calculating torques necessary to pump a well under certain conditions.

A. H. N.

1120.* Pressure Constants Used to Fix Potentials of Flowing Wells. F. B. Taylor. *Oil Wkly*, 1.7.40, 98 (4), 29.—Certain advantages accrue from the use of a restricted flow-pressure-drop relationship for determining actual productivity of wells that do not result from other methods. A full open flow of a producer results in considerable immediate production, and this calls for lease storage or disposal far in excess of normal requirements. By unrestricted open flow of a producer, there is danger of producing a water cone, gas cone, or in some manner bringing about a physical injury to the producing area. Restricted flow tests obviate both dangers. In addition, the pressure decline test can be exactly duplicated a month or a year later, to establish accurately any change or rate of change in the productive ability of a well or field. Correlation through the point at which a well no longer flows to draw down potentials

is possible, so that the change from one method to the other is bridged by fundamental data rather than by supposition. Finally, pressure tests do not require expensive and extensive equipment solely for testing purposes.

The theory of pressure decline tests is very briefly outlined. Two methods are described for determining the index. In general, the well is brought to a steady condition before the test. A pressure bomb is lowered and the pressure is recorded at various depths to determine practically the depth correction in recorded pressures. The well is then made to flow under several rates of production, the maximum being less than 50% of the estimated maximum possibility, and the pressures are recorded under certain steady conditions. By calculation or plotting the result a production index is found.

The paper gives actual requirements and details and shows a specimen calculation by one of the two methods. A. H. N.

1121.* Gravel-Packing Injection Wells for Recycling Gas. H. L. Flood. *Petrol. Engr*, June 1940, 11 (9), 53-54.—Before putting injection wells into operation, in condensate reservoirs, they are under-reamed and gravel-packed in order to obtain low-injection pressures when returning large volumes of gas. A diagrammatic sketch and a detailed account describe the operations involved and general procedure followed in one particular field, where the well is gravel-packed as a routine job in the completion process and not as a separate step taken subsequently as a remedial measure.

After a discussion of the advantages expected to be obtained by using such a procedure in reducing injection pressure requirements generally, it is concluded that data available up to the present are not sufficiently abundant to assess the value of under-reaming and gravel-packing. The gravel-pack is, however, considered, in the least, a worth-while insurance against contingencies that may impair the efficient operation of a gas-injection well. A. H. N.

1122.* Multiple Production from Several Zones Controlled by Bottom-Hole Chokes. W. A. Sawdon. *Petrol. Engr*, June 1940, 11 (9), 109-110.—Allocation of production from the Montebello field, California, is made according to the gas potential of a well when it is completed. Thus with the gas allowable for a well established, the operator strives to produce the maximum of oil with that allowable gas production. As the field has a multiplicity of producing horizons, some rich in gas and poor in oil and others *vice versa*, gas is taken from one zone and passed through chokes to control it and used to lift oil from a deeper formation richer in oil and poorer in gas.

Three types of installations are reproduced diagrammatically and their operations are discussed. The choke is run into the well with running tool jars and sinker bar on a small wire line. When it is in position in the landing nipple it is automatically locked there, and the running tool, sinker bar, and wire line are withdrawn. The choke is removable with a pulling tool, which is also run-in on a wire line. A. H. N.

1123.* Behaviour of Oil and Gas under High Pressures and Temperatures. Part 1. H. T. Kennedy. *Petrol. Engr*, June 1940, 11 (9), 117.—Working on the thesis that analysis of high-pressure gas-oil equilibria may indicate relative position of a well in the reservoir, and thus determine future drilling and proper methods of production, the author details the simple gas laws and then moves on to high-pressure phenomena, and in particular to retrograde characteristics of mixtures of oil and gas under such conditions.

A capillary tube was partly filled with oil of the same concentration as occurred in a particular well under study, and the remainder of the space was filled with gas of the same composition as occurred in the same well. The capillary was sealed and the temperature and pressure were raised to values obtaining in the well. The oil completely disappeared into the gaseous phase. As a control tube containing only oil did not show evaporation of oil without the gas under the same temperature, it was concluded the well penetrated a gas cap under "retrograde" conditions. Wells lower down the structure are producing heavy oil with normal gas-oil ratios, thus justifying the conclusions.

This was in 1930, and is believed to be the first demonstration of wells producing "distillate" oils. A. H. N.

1124.* **Electric Generating Plants on Kansas Producing Properties.** R. A. Axe. *Petrol. Engr.*, June 1940, 11 (9), 132.—Before the adoption of proration regulations in Kansas, the lowest operating costs had been obtained by installing pumping equipment that would pump all the oil a well was capable of producing, thus depleting the well in the shortest possible time and preventing the oil from seeping into an adjoining lease. Even during the initial orders period large-capacity plants were necessary—50-h.p. gas engines driving 40-h.p. pumping units. However, as further orders came into force, making the allowables smaller, smaller units became sufficient, until now only 3- to 4-h.p. units are required. These units are electrical, and the paper gives a description of the general layout and working conditions of the units in the fields showing a material reduction in the operating costs. A. H. N.

1125. **Production Patents.** E. G. Waro. U.S.P. 2,203,759, 11.6.40. Appl. 27.8.38. Pump valve with gas chamber in cage.

F. E. O'Neill. U.S.P. 2,203,796, 11.6.40. Appl. 26.1.39. Method and means of perforating well casing and the like, using laminated members and a jet of abrasive fluid under pressure to form the perforations.

H. C. Glitsch and C. S. Crickmer. U.S.P. 2,203,957, 11.6.40. Appl. 26.4.37. Flow valve adapted to be connected to a tubing string and is pressure-operated.

H. C. Lawton and A. G. Loomis. U.S.P. 2,204,223, 11.6.40. Appl. 30.1.39. Method of treating oil and gas wells for selectively sealing off formations having small quantities of connate waters, these formations being adjacent oil-bearing formations.

D. A. Limerick and H. C. Lawton. U.S.P. 2,204,224, 11.6.40. Appl. 28.2.38. Method for treating oil wells to clean them from high-melting-point organic deposits comprising paraffinic matter by generating heat chemically—i.e. by action of inorganic acid and inorganic oxidizing agent on an electropositive metal.

M. de Groote. U.S.P. 2,204,580, 18.6.40. Appl. 17.2.36. Process for increasing the productivity of wells by treating them with acid containing a wetting agent comprising an alkylated sulpho-aromatic acid.

W. J. Crites. U.S.P. 2,204,725, 18.6.40. Appl. 19.10.37. Means for reducing stresses in oil-well pumping equipment consisting of a compressible medium mounted in the pitman.

J. D. Nixon. U.S.P. 2,204,817, 18.6.40. Appl. 26.7.39. Means for flowing wells comprising a fluid inlet valve attached to a tubing string.

C. V. Temple. U.S.P. 2,204,834, 18.6.40. Appl. 8.10.36. Gas-lift valve comprising a main and a pilot valve, both pressure actuated.

A. Hollander. U.S.P. 2,204,857, 18.6.40. Appl. 13.6.38. Series-parallel submersible pump comprising a submersible motor and multistage pump with valves for selectively connecting stages in series or in parallel.

L. A. Layno. U.S.P. 2,205,421, 25.6.40. Appl. 20.10.36. Means and method of treating wells, comprising a pump, pipe, tank, and valve, and working by filling tank from well and keeping valve closed so that pressure in tank increases, then stopping pump and letting liquid from tank surge into well and thus wash it.

L. A. Layno. U.S.P. 2,205,422, 25.6.40. Appl. 23.12.38. Method for forming a gravel pack in a well bore.

R. A. Wilson. U.S.P. 2,205,739, 25.6.40. Appl. 1.10.37. Gas release valve for wells having a flow line with a surrounding packer.

G. S. Knox. U.S.P. 2,205,748, 25.6.40. Appl. 26.6.37. Well-pipe plug.

J. H. McEvoy and J. H. Howard. U.S.P. 2,205,988, 25.6.40. Appl. 16.7.38. Oil-well equipment, being a well-head completion arrangement. A. H. N.

Transport and Storage.

1126. Transport of Hydrocarbons and of Butane and Propane by Tank-Cars. M. Meot. *Science et Ind.*, 1940, 290 bis (Supplement to *Revue Mécanique*), 108-118.—Rail tank-cars for the transport, through France, of liquid petroleum products and of liquefied butane and propane, together with the portable rail containers for the latter gases, are reviewed. The regulations governing the construction and testing of the tank-cars and containers are surveyed in detail, and several photographs and diagrams illustrate the article. Attention is paid to the methods and regulations for mounting the tanks on to the rail chassis. T. C. G. T.

1127. Handling of Hydrocarbons under an Atmosphere of Inert Gas. H. Pinaud. *Science et Ind.*, 1940, 290 bis (Supplement to *Revue Mécanique*), 99-100.—It is suggested that the reduction of explosion risks by handling and storing inflammable liquids in an inert atmosphere should be further developed. Carbon dioxide is an obvious choice for the inert gas, but exhaust gas from a petrol engine offers good possibilities. If the engine is run primarily to produce an inert gas, an exhaust with a composition of 12% carbon dioxide and 84% nitrogen can be obtained, and such a gas is quite inert from a combustion point of view. A general scheme for handling gasoline whereby during the movement of the liquid it is kept entirely under inert gas control is outlined. T. C. G. T.

Crude Petroleum.

1128. Tabulated Analyses of Texas Crude Oils. A. J. Kraemer and G. Wado. U.S.B. of Mines Tech. Paper 607, 1939.—All analyses of Texas crude oils made by the Bureau of Mines to March 1939 have been tabulated in condensed form. C. L. G.

Cracking.

1129. Catalytic Cracking. Anon. *Science et Ind.*, 1940, No. 290 bis (Supplement to *Revue Mécanique*), 32-35.—A general review of the Houdry catalytic cracking process operated by the Magnolia Petroleum Co. The process is illustrated diagrammatically. Tests of the charging stock and of the gasoline yield are shown and the lead susceptibility of the latter also receives attention. The catalysts are naturally occurring materials such as aluminates and silicates, or oxides of certain metals, such as chromium, tungsten, vanadium, etc. The latter type are, of course, more costly and, though much more effective initially, are more easily poisoned. T. C. G. T.

1130.* Inspection of Cracking Plants. B. F. T. Bruera. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 35-43.—This is a well-illustrated article dealing with corrosion, erosion, super-heating, vibration, and other causes producing failures in cracking plants, and considered from the point of view of the inspector responsible for maintaining them in good condition. The author, in his introduction, states that, in spite of a great deal of experimental work, no alloy has yet been found which will give complete satisfaction in resisting the individual or combined action of temperature, pressure, corrosion, and the like. H. I. L.

1131. Patent on Cracking. H. Tropesch. U.S.P. 2,203,850, 11.6.40. Appl. 24.1.38.—Conversion of hydrocarbon oil by contacting it at cracking temperature with a modified felspar produced by removing from felspar at least a part of its alkalinous base and aluminium content. On the residual portion of the felspar a salt of a metal the oxide of which is catalytic is absorbed, and thereafter calcined to convert the salt into the catalytic metal oxide supported on the residual portion of the felspar. H. B. M.

Hydrogenation.

1132. Patent on Hydrogenation. Harold Edwin Potts. E.P. 522,037, 6.6.40. Appl. 3.12.38.—Process for the destructive hydrogenation of carbonaceous materials and dephenolising of waste waters. The waste waters are injected behind the reaction vessel into the high-pressure cycle of a destructive hydrogenation system. The phenolic substances are thus dissolved in the hydrogenation products and extracted from the water.

H. E. Potts. E.P. 522,254, 13.6.40. Appl. 3.12.38.—Process for the working up of residues of the hydrogenation under pressure of distillable carbonaceous materials. The residues are first subjected to centrifuging and the centrifuged residues are then submitted to filtration at a temperature between 200° and 400° C.

V. Komarowsky. U.S.P. 2,203,826, 11.6.40. Appl. 31.12.37.—Dehydrogenation of aliphatic hydrocarbons by subjecting them to dehydrogenating conditions in the presence of a catalyst comprising a major proportion of alumina and a minor proportion of chromium sesquioxide and nickel.

L. Heard. U.S.P. 2,205,141, 18.6.40. Appl. 31.12.37.—Dehydrogenation of hydrocarbons by contacting them in the vapour phase at a temperature of 650–1050° F. with a metal chromite catalyst. The catalyst is prepared by the thermal decomposition of a crystalline metal ammonium chromate hexahydrate wherein the metallic constituent is one or more metals selected from the group consisting of magnesium, calcium, cobalt, and nickel. H. B. M.

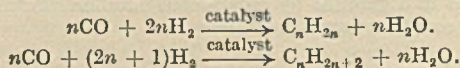
Polymerization.

1133. Patent on Polymerization. Robert Froeborn Ruthuff. E.P. 521,891, 4.6.40. Appl. 11.10.38.—Modification in method of polymerizing olefinic hydrocarbons or mixtures of same. The hydrocarbons are contacted at a high temperature with a solid catalytic agent comprising essentially copper pyrophosphate.

S. C. Fulton and T. Cross. U.S.P. 2,204,673, 18.6.40. Appl. 19.12.35.—Polymerization of normally gaseous hydrocarbons containing olefins by contacting them at a temperature between 200° and 300° C. with a catalyst prepared by impregnating activated silica gel with aluminium chloride dissolved in ethylene dichloride and heating the mass at a temperature sufficient to drive off the ethylene dichloride. H. B. M.

Synthetic Products.

1134.* Industrial Synthesis of Hydrocarbons from Hydrogen and Carbon Monoxide. A. J. V. Underwood. *Industr. Engng Chem.*, 1940, **32**, 449.—This paper gives an up-to-date summary of the Fischer-Tropsch synthesis of hydrocarbons from hydrogen and carbon monoxide, which may be represented by the following reactions:—



The most efficient catalysts for the synthesis are cobalt, nickel, and iron. It is presumed that the metal reacts with the carbon monoxide and hydrogen, forming metallic carbides, which are subsequently reduced by hydrogen with the formation of methylene radicles; these radicles then polymerize to higher hydrocarbons. Various promoters have been used with the catalysts—namely thoria, magnesia, manganese oxide, and alumina—whilst the most satisfactory support is kieselguhr. In the latest industrial units a supported cobalt catalyst is employed because this metal gives the greatest yield of hydrocarbons and has the longest life. The theoretical yield of hydrocarbons from 1 cubic metre of ideal gas (2 parts H₂ plus 1 part CO) is 208 gm.; yields of 130–140 gm. have been realized in one-stage commercial operation. The synthesis is

carried out at temperatures in the range 190–200° C. and at pressures of from 5 to 10 atmospheres. Increase of pressure has a marked effect on the yield and character of the product, increase of pressure bringing about an increase in yield until a point is reached when further increase in pressure decreases the yield. For operating pressures in excess of 5 atmospheres there is a decrease in the formation of hydrocarbons boiling in the gasoline range, and an increase in the formation of wax. Pressure operation increases catalyst life, and the total yield of hydrocarbons decreases less rapidly than under atmospheric pressure conditions. The activity of the catalyst decreases to some extent by the deposition of wax on its surface, although a high activity can be maintained by periodic removal of the wax, which may be accomplished by treating the catalyst with a suitable solvent or with hydrogen. Accumulated data support the theory that contact time is not a controlling factor in the process, but is rather the rate at which the reaction products can be removed from the neighbourhood of the catalyst.

The industrial process as used by the Ruhrchemie A.-G. is described in outline.

H. E. T.

Refining and Refinery Plant.

1135. Review of the Literature on the Construction, Testing and Operation of Laboratory Fractionating Columns. C. C. Ward. U.S.B. of Mines Tech. Paper 600, 1939.—The report includes: (1) a general consideration of fractionating unit design and operation, (2) the evaluation of columns with examples of graphical and analytical methods of computing the number of theoretical plates, and vapour liquid equilibria data for some common binary test mixtures, (3) a discussion of details of the construction, operation, and evaluation of various fractionating units, and (4) conclusions as to the principal factors to be considered when designing an efficient fractionating unit.

C. L. G.

1136.* Extraction of Mercaptans with Alkaline Solutions. D. L. Yabroff. *Industr. Engng Chem.*, 1940, **32**, 257.—The factors affecting the distribution of a mercaptan between an oil phase and an aqueous alkaline solution are discussed. Data are presented illustrating the distribution of the mercaptans up to heptyl mercaptan between *iso*-octane and aqueous phases of varying sodium hydroxide concentration at 20° C. With increasing molecular weight the solubility of the mercaptans in water falls rapidly. The differences in the extractability of various mercaptans from an oil phase by an aqueous alkaline solution are due almost entirely to differences in the solubility of the unneutralized mercaptan in the aqueous phase.

With increasing sodium hydroxide concentration the improvement in the extraction is less than might be expected from the higher caustic concentration due to the salting out of the unneutralized mercaptan from the aqueous phase. The effect of temperature over the range 0–50° C. on the hydrolysis and partition of *n*-butyl mercaptan between *iso*-octane and aqueous phases has been examined. The extraction of the mercaptan increases as the temperature is lowered, because of the decreased hydrolysis of the mercaptide in the aqueous phase.

Equations, illustrated by an example, are given enabling the extent of the extraction, of mixed mercaptans in a gasoline by an aqueous alkaline solution, to be calculated under once through counter-current conditions.

J. W. H.

1137.* Mixtures of Ketones Used to Dewax Motor Lubricants. A. J. Mueller. *Oil Gas J.*, 1944, **38** (49), 54.—Commercial operation of a 2000-bbl.-per-day solvent dewaxing plant at a Sarnia (Canada) refinery has confirmed the superior qualities of a mixture of methyl *n*-butyl ketone and methyl *n*-propyl ketone. The latter material has the advantage over the former in lower solubility for wax, higher filtering rate, and lower cost, and gives an oil of pour point below the dewaxing temperature, but has a higher miscibility temperature with oil, a lower vapour pressure, and a higher solubility in water. These disadvantages are reduced by blending with methyl *n*-butyl ketone, by providing adequate condensing equipment, and by deketonizing waste water.

Production data on dewaxing Mid Continent and Loudon (Illinois) distillate indicate a filter rate of 6 gals. dewaxed oil per sq. ft. per hr. with solvent to oil ratios of less than 2 to 1. Pour points on some stocks are 16° F. below filtration temperature. The wax cake (yield about 10%) is washed to an oil content of 6-8%, simplifying subsequent sweating and increasing the yield of wax. Average solvent loss is 0.1%; separating the cycle-wash solvent from the oil-solvent filtrate and employing it for diluting the incoming charge reduces the quantity of solvent to be recovered and prevents contamination of the filtrate with wax dissolved in the wash solvent. The diluted charge is cooled by controlled chilling from 90° to 30° F. and then shock cooled. No filter aid or precoat is necessary in the filters. No toxic or inflammability effects have been encountered.

C. L. G.

1138.* Cathodic Protection of Condensers and Coolers Utilizing Brackish Bay Water. W. A. S. Wright and J. H. Brooks. *Oil Gas J.*, 30.5.40, 39 (3), 60.—Rapid failure of red brass condenser tubes used with brackish bay water for condensing exhaust steam in a San Pablo Bay (Calif.) refinery has led to the following measures: Replacement by Admiralty metal tubes, installation of cathodic protection equipment, use of strainers in salt-water line and a more rigid inspection and cleaning programme.

As a result there has been no tube failure after 14 months' service, only slight corrosion and some scale deposition. Owing to the combination of changes it is not possible to consider the relative effect of the cathodic protection alone. This consisted of cast-iron anodes, 2.5 in. thick, installed in each of the four doors of the condenser, using an electrical current of 7 amps. at 4½ volts, corresponding to 2½ amps. per 1000 sq. ft. of surface. The electrodes are decomposed at the rate of 35 lb. per amp. year, installation being arranged to facilitate replacement. The mild pitting which has been experienced may be due to spacing the anodes too close to the tube sheet, with the resulting turbulence liberating air dissolved in the circulating salt water. The scale deposition has an insulating effect, and results in the current being carried further into the tubes.

Further cathodic protection units have been installed on fresh water-coolers, no tube failures having been reported after 16 months.

C. L. G.

1139.* Corrosion in Refineries. C. Lucero. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 30-34.—This article deals with the various acids which result in corrosion of the tubes and other parts of the distilling plant. Of these hydrochloric acid and sulphur compounds are responsible for the bulk of the damage, the latter compounds being the most active agents at high temperatures. Methods of combating the corrosion by the use of soda and ammonia are described.

H. I. L.

1140.* Prevention of Accidents in Refineries. R. Galletti. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 44-52.—This article deals very fully with conditions in refinery plants with respect to accidents and sickness caused directly by the conditions under which the men have to work and by the materials which have to be used in the processes connected with the petroleum industry.

H. I. L.

1141. Petroleum Refineries, including Cracking Plants in the United States. G. R. Hopkins and E. W. Cochrane. U.S. Bur. Mines. Information Circular No. 7124, Jan. 1940.—On 1st January, 1940, there were nine more completed refineries in the United States than on 1st January, 1939. This is the first time for four years that the Bureau of Mines has been in a position to record an increase in the number. Actually the total completed on 1st January, 1940, was 547, but eighty-six of these were shut down and some of the rest operating only in part. Nevertheless the figure for operating plants was the highest ever recorded, and seven greater than the previous record established in January 1940.

Practically all the gains in operating plants during 1939 occurred in the Central States, where there were significant oil discoveries.

On 1st January, 1940, the operating capacity of crude petroleum plants was recorded as 4,196,694 brls. and reflected a substantial increase over the 1939 figure. In the same way the idle capacity fell from 574,770 brls. to 431,952 brls. during this period.

New construction continued to decline as far as crude oil capacity was concerned,

and, in addition, the nature of new construction changed in 1939, most of it being additional, whereas in 1938 the greater part was constructed for replacements.

On 1st January, 1940, capacity of active cracking units was recorded as 964,555 brls. per day, and was 10% greater than the figure for the previous year. Idle cracking capacity declined in 1939, but, unlike crude oil, the capacity of cracking units under construction was somewhat higher than the year before. Daily output of cracked gasoline in 1939 was 809,000 brls.—i.e., 88% for active units or 79% for both active and inactive plants.
H. B. M.

1142. Manufacture and Use of Petroleum Products. A. J. Kraemer and H. M. Thorne. U.S. Bur. Mines Reprint, 1940.—In 1934 these authors presented a report to a sub-committee of the Committee of Interstate and Foreign Commerce on the effect of technologic factors on the supply of and demand for petroleum products. The present report represents an endeavour to bring the earlier discussion up to date and to direct attention to changes in sources and character of raw materials and products.

It is divided into five sections. Section 1 concerns production of crude petroleum in the United States and gives data relating to sources and stocks of raw materials of the refining industry. The oils vary from very volatile to those which are practically solid at 75° F., and no two crudes are exactly alike. For convenience, however, they are classified into groups according to production areas.

Section 2 includes data regarding the number of refineries in the United States and their distribution among the ten refinery districts.

Information is given on the number of refineries in operation, the number shut down, and total refining capacity for each district; also similar data on cracking plants. In addition, lists are given of quantities of raw materials used and what was made from them, thus indicating the influence of supply and demand on refinery practice in the different districts.

The third section of the report is devoted to petroleum refining technology. Processes of hydrogenation, cyclization, aromatization, and isomerization are briefly described, and emphasis is laid on the value of new refining methods which utilize inferior or waste products to produce superior fuels.

Section 4 comprises a review of certain technologic factors which affect the supply of and demand for the principal petroleum products. Possibilities of increasing supplies of products should the necessity arise and significance of changes in demand for specific products are also discussed. Throughout the section it is borne in mind that almost all petroleum products are used by burning them and that individuals rather than industries are the principal consumers.

The final section of the report deals with the possibilities of alternative fuels being obtained from coal, oil shale, and alcohol. It is pointed out that in each case a greater expenditure in labour and materials is involved than in the manufacture of motor fuels from petroleum.
H. B. M.

1143.* Dehydration and Desalination of Oil at Plaza Huincul. H. M. Gonzalez. *Bol. Inform. Petroleras*, Jan. 1940, XVII (185), 24-28.—This article describes experimental treatment of oil at Plaza Huincul for removal of water and salt. The plant is described and a table of results given. These are fairly satisfactory, and the saving on transport is shown as \$16,770.
H. I. L.

1144.* Problem of Dehydration and Desalination of the Petroleum in Comodoro Rivadavia. C. A. Delorme. *Bol. Inform. Petroleras*, Jan. 1940, XVII (185), 11-23.—This article develops primarily theoretical and experimental data on the question of emulsions, and deals further with the petroleum emulsions of the Comodoro Rivadavia zone, their dehydration on commercial lines, existing dehydration plant, their mode of operation, the effect of other problems and of future requirements. Some interesting photomicrographs are included with the illustrations.
H. I. L.

1145. Patents on Refining. W. A. Schulze. U.S.P. 2,204,234, 11.6.40. Appl. 9.11.38. Process for sweetening and stabilizing petroleum oils by admixing air with them, percolating the admixture through a solid absorbent material consisting of fuller's earth impregnated with a solution of a soluble copper salt and a soluble chloride

salt. Thereafter the mixture of oil and unreacted air is contacted with a solid absorbent material consisting of fuller's earth impregnated with an aqueous solution of a non-oxidizing acid compound insoluble in the oil.

A. W. Hixon. U.S.P. 2,204,758, 18.6.40. Appl. 17.12.36. Refining of paraffinic and non-paraffinic petroleum oils by contacting with furonitrile at temperatures above the miscibility temperature, cooling the mass to stratify it into an oil phase and a solvent phase. Thereafter the solvent phase is separated out and treated with triamylamine under conditions designed preferentially to extract certain of the oil constituents from the furonitrile phase.

D. S. McKittrick. U.S.P. 2,204,003, 18.6.40. Appl. 20.7.38. Desulphurizing low-boiling mineral oil by contacting it in the liquid state at about 40° C. with a refining agent comprised essentially of an aliphatic sulphonic acid containing not more than 5 carbon atoms and being liquid at 40° C. The process is carried out under conditions which cause the formation of a sulphonic-acid phase containing absorbed sulphur compounds and a liquid-oil phase. The two phases are finally separated.

R. D. Howard. U.S.P. 2,205,410, 25.6.40. Appl. 25.3.36. Refining of crude mineral oil distillates which are relatively low boiling and contaminated with mercaptan sulphur compounds. The hot vapours of the distillate are mixed with a small portion of a chlorinating agent prediluted with an oxygen-containing gas compatible therewith. The resultant mixture is contacted with a dechlorinating material containing zinc in oxidized form to dechlorinate and sweeten the vapours and a refined relatively low-boiling mineral oil distillate is condensed from the residual vapours.

Edelenau Gesellschaft m.b.H. E.P. 521,658, 28.5.40. Appl. 24.11.38. Extraction of mineral oils, mineral oil fractions, or residual oils with selective solvents. The oil to be treated and the solvent are led in counter current to each other, and the oil is cooled before entering the extraction apparatus. A portion of the extract solution is withdrawn from the extraction apparatus and mixed with the oil before it enters the cooling apparatus affixed to the front of the extraction apparatus. H. B. M.

Chemistry and Physics of Petroleum.

1146. Isolation of 3-Methylhexane, *trans*-1 : 2-Dimethylcyclopentane and *trans*-1 : 3-Dimethylcyclopentane from Petroleum. A. R. Glasgow, Jr. *Bur. Stand. J. Res. (Wash.)*, 1940, 24 (5), 509-529.—The fraction of a mid-continent petroleum, b.pt. 90-92° C., previously freed from aromatic hydrocarbons, was used for this work.

Systematic distillation of this material showed that naphthenic compounds were concentrating below 90° C., paraffins in the region 90-91.2° C., and the volume of distillate fell to a minimum at 91.2° C. with increasing naphthenic content.

The portion b.pt. 90-91.2° C. was processed by azotropic distillation with methyl alcohol, which resolved it into paraffinous and naphthenic concentrates.

From the naphthenic concentrate *trans*-1 : 3-dimethylcyclopentane was isolated by crystallization from liquid ethane.

The fraction b.pt. 91.2-92.2° C. was then distilled with methyl alcohol alternating with ordinary distillation, and *trans*-1 : 2-dimethylcyclopentane was isolated by crystallization from liquid propane plus methane. From the paraffinic concentrate 3-methylhexane was obtained in the residue from distillations at 315 mm. Hg.

The b.pt., refractive index, f.pt., density, C/H ratio, and aniline point were determined for the three hydrocarbons. Regarding the content of *n*-heptane as 1, the relative amounts of the three hydrocarbons in this petroleum were estimated to be : *trans*-1 : 3-dimethylcyclopentane 0.21; 3-methylhexane 0.25; *trans*-1 : 2-dimethylcyclopentane 0.31.
D. L. S.

1147. Vapour-Liquid Equilibrium. V. Carbon Tetrachloride-Benzene Mixtures. G. Scatchard, S. E. Wood, and J. M. Mochel. *J. Amer. chem. Soc.*, 1940, 62, 712-716.—This work completes the measurements on binary systems of the compounds carbon tetrachloride, cyclohexane, and benzene. As before (*J. Amer. chem. Soc.*, 1939, 61, 3206-3210; *J. Inst. Petrol.*, 1940, 26, Abstr. No. 651) the vapour-liquid

equilibrium pressure and composition were measured at 40° and 70° C. throughout the composition range of carbon tetrachloride and benzene mixtures, and at 30°, 50°, and 60° C. for approximately equimolar mixtures. The densities were measured at 25° C. Analysis of these results shows slight deviations from corresponding equations derived from thermodynamic functions, including the energy and entropy of mixing at constant total volume. Approximate equations are derived for the ternary system benzene-cyclohexane-carbon tetrachloride. R. D. S.

1148. Attempt to Synthesize a Substituted *cyclo-Octatetraene*. S. Wawzonok. *J. Amer. chem. Soc.*, 1940, **62**, 745-749.—An attempt was made to synthesize *sym.*-dibenzocyclooctatetraene from diphenylsuccinide-10 through *sym.*-dibenzocyclooctanedione-5, 11, but as the synthesis could not be completed, the properties of the cyclic diketone were examined and found to be similar to those of open-chained 1:5-diketones. R. D. S.

1149. Separation of the Isomeric Hexenes by Batch Fractionation. A. Roso. *J. Amer. chem. Soc.*, 1940, **62**, 793-795.—This paper applies an extension of the reasoning of Sidney Young and of Rayleigh to the separation of the isomeric hexenes in an attempt to arrive at the required fractionating efficiency and some idea of the results to be expected. The computations show that even with practically total reflux and zero hold-up at least 400 theoretical plates are required to ensure sharp separation of some of the close-boiling hexenes. R. D. S.

1150. Attempted Separation of the Isomeric Hexenes by Fractional Distillation. F. C. Whitmore, and others. *J. Amer. chem. Soc.*, 1940, **62**, 795-800.—The authors were unable to substantiate work on this subject by Goldwasser and Taylor (*J. Amer. chem. Soc.*, 1939, **61**, 1751), who employed a Podbielniak-Simons-Taylor column, for whereas the investigations of Goldwasser and Taylor indicated separation of six hexenes with a total b.pt. range of 2.5° C., the present authors could not obtain separations even with two binary mixtures of purer hexenes of boiling ranges of 1.5° C. and 2.7° C., or with a ternary mixture having a b.pt. spread of 2.7° C.; furthermore, whilst the Podbielniak-Simons-Taylor column has an efficiency of only about fifteen theoretical plates, the calculations in the previous paper show that an efficiency of more than 400 theoretical plates is necessary to effect separations of the six hexenes. R. D. S.

1151. Vapour Pressures of Certain Unsaturated Hydrocarbons. A. B. Lamb and E. E. Roper. *J. Amer. chem. Soc.*, 1940, **62**, 805-814.—Using a double-vapour-pressure apparatus permitting the simultaneous measurement of the vapour pressures of two samples at a given temperature-setting of a surrounding cryostat, the authors have measured the vapour pressures of liquid ethene, propene, 2-methylpropene, 1-butene, *cis*-2-butene, *trans*-2-butene, 1:4-pentadiene and 2-methyl-2-butene over the approximate range 10-1000 mm., and have derived by the method of least squares from these results equations containing three constants expressing vapour pressure as a function of temperature; they have also obtained constants for an empirical equation for propadiene in the crystalline phase by drawing the best line through the data and constants for the equation for liquid propadiene by the least-squares method. From the empirical equations for the vapour pressure the authors have computed the boiling points and temperature coefficients of the vapour pressures, and from these, in turn, obtained the molecular heats of vaporization. Generally, the vapour pressures quoted show satisfactory agreement with the Ramsay-Young rule and the Dühring rule, especially where in each rule an additional constant is introduced. A general equation has been derived which expresses the temperature differential of the vapour pressure at the absolute boiling point as a function of the absolute boiling point. R. D. S.

1152. Halogenation of Unsymmetrical Diphenylethane. F. E. Shibley and C. F. Prutton. *J. Amer. chem. Soc.*, 1940, **62**, 840-841.—When the product of the photochemical chlorination of 1:1-diphenylethane is distilled the distillate consists of unchanged diphenylethane, stilbene, and 1:1-diphenyl-2:2-dichloroethylene. Bromination mixtures, similarly prepared and distilled, give only small amounts of

1 : 1 : 4 : 4-tetraphenylbutadiene-1 : 3 and stilbene. Owing to its remarkable stability, 1 : 1-diphenyl-2 : 2-dichloroethylene requires the action of alcoholic caustic potash under pressure at 150° C. to effect complete hydrolysis. The pyrolysis of 1 : 1-diphenylethane in a silica tube at 700° C. gives benzene, toluene, and styrene in small amounts.

R. D. S.

1153. Condensation of Esters with Aromatic Hydrocarbons by Means of Aluminium Chloride. J. F. Norris and P. Arthur, Jr. *J. Amer. chem. Soc.*, 1940, **62**, 874-877.—This paper presents results of investigations into the condensations of methyl and ethyl formate and acetate with benzene and toluene by means of aluminium chloride, with reference to the conditions necessary to produce alkylation alone or simultaneous alkylation and acylation. It was found that alkylation and acylation required a higher temperature than alkylation alone for ethyl acetate; acylation of methyl and ethyl formates could not be effected. The pyrolytic product of methyl acetate and aluminium chloride produced methyl chloride, and the residue obtained in a like manner from ethyl acetate and aluminium chloride formed acetophenone in 42% yield when it was condensed with benzene. Identification of the ketones formed from methyl acetate was performed by use of some new 4-nitro- and 2 : 4-dinitro-phenylhydrazones of 2 : 4- and 2 : 5-, and 3 : 4-dimethylacetophenones prepared by the authors.

R. D. S.

1154. 9-Methyl-3 : 4-Benzfluorene. L. F. Fieser and Ll. M. Joshel. *J. Amer. chem. Soc.*, 1940, **62**, 957-958.—This compound was synthesized because of the somewhat analogous hydrocarbon 10-methyl-1 : 2-benzanthracene being of marked carcinogenic activity. 3 : 4-Benzfluorenone-9 was prepared from 1-phenyl-2 : 3-naphthalic anhydride and the ketone was then condensed with methyl magnesium chloride to a carbinol which gave the desired hydrocarbon on heating in acetic acid and shaking with hydrogen and Adams catalyst.

R. D. S.

1155. Synthesis of 2-Methyl-3 : 4-Benzphenanthrene. M. S. Newman and Ll. M. Joshel. *J. Amer. chem. Soc.*, 1940, **62**, 972-974.—Direct cyclization of *p*-benzohydroxyglutaric acid using anhydrous hydrogen fluoride, or ring closure of the anhydride using aluminium chloride, gave 1 : 2 : 3 : 4-tetrahydro-4-keto-1-phenyl-2-naphthalene-acetic acid. The reduction of the keto-group gave the hexahydro-ketone, which, under the influence of the appropriate Grignard reagent, followed by dehydration and dehydrogenation, gave 2-methyl-3 : 4- and 2-ethyl-3 : 4-benzphenanthrene. The former of these hydrocarbons is very carcinogenically potent when painted on the skin, but not so active when applied by the injection technique.

R. D. S.

1156. Low Temperature Dehydrogenations. II. R. T. Arnold, C. Collins, and W. Zenk. *J. Amer. chem. Soc.*, 1940, **62**, 983-984.—A note on the preparation of unsymmetrical biphenyl, terphenyls, etc., in 50-70% yields by dehydrogenation of substituted cyclohexenes under reflux with chloranil or other quinones with high oxidation potentials.

R. D. S.

1157. Viscosities of Linear Poly-esters. An Exact Relationship Between Viscosity and Chain Length. P. J. Flory. *J. Amer. chem. Soc.*, 1940, **62**, 1057-1070.—The author has measured the viscosities, densities, and thermal expansion coefficients of linear poly-esters having molecular weights ranging from 200 to over 10,000, and finds that the logarithm of the viscosity is an exact linear function of the square root of the weight average chain length or of the square root of the weight average molecular weight according to the equations: $\log \eta = A + C'M_w^{\frac{1}{2}} = A + CZ_w^{\frac{1}{2}}$, where M_w is the weight average molecular weight, Z_w the weight average chain length, and A , C , and C' are constants.

The relationship between viscosity and weight average chain length applies to mixtures of two poly-esters of low and high average molecular weights, respectively; hence the author deduces that it is independent of the type of distribution of species in the polymer. The molecular weight has only a slight influence on the viscosity-temperature coefficient, so that the relationship between viscosity and weight average

chain length, as well as the constant C in the equation, must likewise be independent of temperature. The author concludes that the elementary process of viscous flow must consist either of a displacement of a small portion of the entire molecule or of a displacement of the entire molecule through a comparatively small distance; furthermore, such a displacement cannot be commensurate with the size of the molecules, and the activation energy for viscous flow must be unconnected with molecular size. The equation can be further usefully employed to determine directly the weight average molecular weight from viscosities of the poly-esters. R. D. S.

1158. Petroleum Bases. I. Reactions of 2:3:8-Trimethylquinoline. A. Burger and L. R. Modlin. *J. Amer. chem. Soc.*, 1940, **62**, 1079-1083.—A study of the reactivity of the 2 and 5 positions of the quinoline system of this kerosine base. R. D. S.

1159. Redistribution Reaction. V. R,Pb Compounds. G. Calingaert, H. A. Beatty, and H. Soroos. *J. Amer. chem. Soc.*, 1940, **62**, 1099-1104.—The redistribution reaction is perfectly general for these compounds, as data on fourteen systems, both alkyl and aryl, show. The effect of such factors as the R groups, catalysts, catalyst concentration, solvents, temperature, and time are given. R. D. S.

1160. Redistribution Reaction. VI. Alkyl Lead Halides. G. Calingaert, H. Soroos, and H. Shapiro. *J. Amer. chem. Soc.*, 1940, **62**, 1104-1107.—From the data presented in this paper the authors confirm that when trialkyl-lead halides undergo the redistribution reaction they require no other catalyst than themselves. Six systems were investigated, comprising a mixed trialkyl-lead halide, four trialkyl-lead halide-tetraalkyl-lead systems, and one system of two different trialkyl-lead halides, the alkyl groups being methyl and ethyl. For systems involving only trialkyl-lead halides the theory predicts a random equilibrium mixture of all four possible trialkyl-lead halides; for systems involving tetraalkyl-lead, the theory predicts five lead alkyls and four trialkyl-lead halides. These random equilibria were confirmed by analysis after completion of reaction. R. D. S.

1161. Redistribution Reaction. VII. Alkyl Compounds of Mercury, Tin, Silicon, and Zinc. G. Calingaert, H. Soroos, and V. Hrizda. *J. Amer. chem. Soc.*, 1940, **62**, 1107-1110.—The results of these experiments demonstrated that mercury, tin, and silicon alkyls undergo the redistribution reaction in the same way as these compounds of lead. Similar zinc compounds, however, did not react, even with catalysts, such as aluminium chloride at the boiling temperature (60° C.); different conditions and higher temperatures have not yet been applied. Contrary to previous reports in the literature, methyl ethylmercury was found to be stable on distillation or on standing, when free from catalytic amounts of alkylmercuric chloride which causes redistribution. R. D. S.

1162. Thermodynamics of *n*-Heptane and 2:2:4-Trimethylpentane, including Heat Capacities, Heats of Fusion, and Vaporization and Entropies. K. S. Pitzer. *J. Amer. chem. Soc.*, 1940, **62**, 1224-1227.—The thermal characteristics were investigated between 15° and 423° K., and tables in the paper give the heat capacities in the solid, liquid, or gas phase. The other properties are:—

		<i>n</i> -Heptane.	2:2:4-Trimethylpentane.
Heat of fusion,	cal. per mole.	3355.8 ± 4	2201.6 ± 2
Heat of vaporization	" "	7660 ± 20	7410 ± 20
Entropy (liq., 298.1° K.)	" "	78.60 ± 0.2	78.40 ± 0.2
Entropy (gas, b.pt.)	" "	111.77 ± 0.3	112.05 ± 0.3

R. D. S.

1163.* Temperature of Flame Gases. W. T. David. *Engineering*, 1940, **149**, 241.—The calculated and experimentally determined flame temperatures for propane and ethylene are compared over a range of mixture strengths. The experimental determinations were those by the sodium-line reversal method. The various possible reasons for the discrepancies between the two sets of values are reviewed, and it is

suggested that a possible reason is that the gas mixtures were not of uniform composition. The conclusion is reached that sodium-line reversal determinations are untrustworthy, and that some form of wire method (resistance or thermocouple) is preferable.
P. D.

1164.* Calculation of Chemical Equilibrium at High Pressures. R. H. Ewell. *Industr. Engng Chem.*, 1940, **32**, 2, 147.—The methods of calculating equilibrium from available thermal data are discussed and their application is illustrated by calculations of the methanol synthesis, the hydration of olefins, and the production of hydrogen cyanide from nitrogen and acetylene.
P. D.

1165.* Detergents from Kerosine. A. R. Padgett and E. F. Degering. *Industr. Engng Chem.*, 1940, **32**, 204.—A kerosine fraction boiling at 95–100° C. (15 mm.) was chlorinated to give a yield of 80–90% of monochlorides. Conversion of the monochlorides into the corresponding alcohols was accomplished by a procedure essentially that employed by the Sharples Solvents Corporation, the yield being about 30%. The alcohols were then converted into the corresponding sodium alkyl sulphates.

The 2-, 3-, 4-, 5-, and 6-dodecanols were prepared and the corresponding alkyl sulphates were obtained from them; sodium lauryl sulphate was also prepared from 1-dodecanol.

The various sodium alkyl sulphates were compared by means of foam tests and an interfacial tension test. These tests indicated that the ability to lower interfacial tension and the foam value for a given chain decrease progressively from a maximum for a primary to a minimum for a symmetrical secondary sodium alkyl sulphate.

The sodium alkyl sulphates prepared from the kerosine fraction are inferior to similar compounds with a terminal polar group when assessed by the foam and interfacial tension tests.
H. E. T.

1166.* Evaporative Index. G. S. Gardner. *Industr. Engng Chem.*, 1940, **32**, 226.—The mathematical derivation of a simplified Maxwell-Stefan equation is given for the rate of evaporation (E) at a definite point on a liquid surface under constant temperature, pressure, and air flow conditions as

$$E = K(D_1 M p_{A1})$$

in which the group ($D_1 M p_{A1}$) termed the evaporative index may be calculated from a knowledge of the diffusion coefficient (D_1), the molecular weight (M), and the vapour pressure of the evaporating liquid (p_{A1}). For the purpose of the above equation it is sufficient to calculate the diffusion coefficient from the molecular volume by means of the Gilliland equation. The practical utility of the evaporative index is demonstrated by a comparison between calculated and determined values of the rate of evaporation, under carefully controlled conditions, for a large number of different liquids.

The term K in the above equation equals $\frac{\alpha}{RTpx}$, where α is a proportionality constant and x the thickness of the stagnant film above the surface of the liquid. By assuming that x varies as a function of the Reynolds group, it is possible to extend the simplified equation to apply to varying conditions of temperature and pressure.
J. W. H.

1167.* Catalytic Dehydrogenation of Gaseous Paraffins. A. V. Grosse and V. N. Ipatieff. *Industr. Engng Chem.*, 1940, **32**, 268.—A catalytic dehydrogenation process has been developed for converting normal and *isobutane*, propane and ethane into the corresponding olefins. The process consists essentially in passing the preheated paraffin gases over a suitable catalyst. The outgoing gases contain olefins and hydrogen besides unreacted paraffin. The olefins are polymerized or used in alkylation reactions, the hydrogen is separated and the residual paraffins are recycled. A suitable catalyst for an economic process must satisfy the following conditions:—

- (1) It must have the ability to selectively split off hydrogen without cleavage of C-C bonds.
- (2) It must be easily regenerated when fouled by carbon.
- (3) It must have a comparatively long life.
- (4) It must be low-priced.

The above requirements are fulfilled by selected solid catalysts containing small quantities of the oxides of the transition metals of groups VI (chromium and molybdenum), V (vanadium), and IV (titanium and cerium) of the periodic system, supported on carriers of relatively low activity (e.g., aluminium and magnesium).

Under proper dehydrogenating conditions over-all conversions of the paraffin into olefins of 90–95% of theoretical were obtained, and at the same time an equal volume of almost pure hydrogen (over 90%) suitable for catalytic hydrogenations was produced as a side-product.

The olefins produced were identified by molecular weight, boiling point, refractive index, and formation of the bromides.

The dependence of conversion of paraffins into olefins on space velocity, temperature, and age of catalyst is discussed.
H. E. T.

1168.* Benzene and Toluene Isopiestic Liquid-Vapour Equilibrium Data. F. Todd. *Industr. Engng Chem.*, 1940, **32**, 2, 287.—The data on the benzene-toluene system in the literature is critically examined and a table giving the equilibrium compositions on boiling points at 760 mm.
P. D.

1169.* Free Discharge of Fluids through Small Circular Orifices. H. L. Roy and N. K. Sen-Gupta. *Industr. Engng Chem.*, 1940, **32**, 2, 288.—Experiments were made on the free discharge from a large tank through orifices ranging from $\frac{1}{8}$ to $\frac{3}{8}$ in. in diameter, using water and oils with viscosities up to 700 centipoises. The Reynolds number was varied from 1 to 10^5 . The results are presented in tabular and graphical form.
P. D.

1170.* Catalytic Alkylation of *iso*Butane with Gaseous Olefins. F. H. Blunck and D. R. Carmody. *Industr. Engng Chem.*, 1940, **32**, 328.—Alkylation of *isobutane* at 1000 lb. per square inch pressure and at a temperature of 400° F. has been effected with the following olefins, ethylene, propylene, and *isobutylene*, under the catalytic influence of the double chlorides of aluminium and alkalic metals. The double compounds of alkalic halides and aluminium halides used as catalysts were supported on pumice, which was shown to have no catalytic action. The alkylation reaction is accompanied by a varying but considerable amount of polymerization and by extensive rearrangements, leading to the production of side products which are difficult to explain by any simple theoretical consideration. Catalyst life is of short duration, and the rate of decay of the alkylation reaction is greater than that for polymerization. High temperatures favour alkylation, but at the same time produce a decrease in catalyst life.
H. E. T.

1171.* Liquid-Vapour Equilibrium Relations in Binary Systems. W. B. Kay. *Industr. Engng Chem.*, 1940, **32**, 3, 353.—The P-V-T-x relations were measured for the ethane-*n*-butane system over the entire range up to the critical. The results are given in graphical and tabular form. Equilibrium constants were derived from the results and are compared with those obtained by calculation. The equilibrium constants for ethane in *n*-butane are compared with those for ethane in *n*-heptane.
P. D.

1172.* P-V-T Relations for *n*-Butane. W. B. Kay. *Industr. Engng Chem.*, 1940, **32**, 3, 358.—The vapour pressure and density of the saturated liquid and vapour were measured from room temperature up to the critical point. The compressibility of liquid and vapour was measured from 100° F. to 600° F. and 35 to 1200 lb. per sq. in.
P. D.

1173.* Specific Heat Ratios for Hydrocarbons. W. C. Edmister. *Industr. Engng Chem.*, 1940, **32**, 3, 373.—The C_p/C_v ratios for seventeen hydrocarbons were plotted on a reduced basis. It was found that the plots could be made to coincide by using a single multiplying factor for each hydrocarbon. Using this as a basis, a chart is presented from which the value of the ratio for any one of the seventeen hydrocarbons can be obtained at reduced pressures up to 1.2 and reduced temperatures up to 2.5.
P. D.

1174.* **Phase Equilibria in Hydrocarbon Systems. Joule-Thomson Coefficients for Gaseous Mixtures of Methane and *n*-Butane.** R. A. Budonholzor, B. H. Sage, and W. N. Lacey. *Industr. Engng Chem.*, 1940, **32**, 3, 384.—Joule-Thomson coefficients were measured for five mixtures at temperatures between 70° and 310° F. and pressures between 50 and 1500 lb. per sq. in. The figures obtained, combined with the isobaric heat capacity at infinite attenuation, are used to calculate the heat capacities of several mixtures as functions of pressure and temperature. The results are presented in graphical and tabular form. P. D.

1175.* **Isothermal Changes in Enthalpy for Some Gases.** R. York and H. C. Weber. *Industr. Engng Chem.*, 1940, **32**, 3, 388.—Isothermal changes of enthalpy with pressure of ethane, propane, butane, and heptane have been correlated by an empirical method. The correlation is compared with values taken from the literature for carbon dioxide, methane, and benzene. The method does not fit the gases of low-boiling liquids, nor the so-called highly polar compounds, but it is believed to be reliable between $Tr = 1.00$ and $Tr = 1.80$ and $Pr = 0$ and $Pr = 8$ for hydrocarbons with critical temperatures between 300° and 600° K. P. D.

1176.* **Analytical Calculations of Thermodynamic Properties.** H. M. Robinson and H. Bliss. *Industr. Engng Chem.*, 1940, **32**, 3, 396.—Equations for calculating entropy and enthalpy in terms of volume have been derived for the Van du Waals, Wohl, and Beattie-Bridgeman equations of state, and have been tested by comparing calculated values of entropy and enthalpy with those determined graphically. The Beattie-Bridgeman equation is satisfactory to 5-7% for entropy and to 12% for enthalpy. P. D.

1177.* **Sulphonation and Nitration Reaction Promoted by Boron Fluoride.** R. J. Thomas, W. F. Anzilotti, and G. F. Hennion. *Industr. Engng Chem.*, 1940, **32**, 408.—Boron fluoride is an effective promoter and dehydrating agent for many sulphonation and nitration reactions. Experimental details are given of typical processes involving the use of boron fluoride. By the use of boron fluoride it is possible to sulphonate or nitrate with the stoichiometric amounts of sulphuric or nitric acid; the boron fluoride is converted into the monohydrate and, if an excess of water be added, the dihydrate is formed from which the anhydrous boron fluoride may be recovered. J. W. H.

1178.* **Nitration of Ethane.** H. J. Hibshman, E. H. Pierson, and H. B. Hass. *Industr. Engng Chem.*, 1940, **32**, 427.—The trinitrate of 2-hydroxymethyl-2-nitro-1:3-propanediol and the dinitrate of 2-methyl-2-nitro-1:3-propanediol are high-explosive derivatives of nitromethane and nitroethane. These products may be produced from air, steam, and natural gas as raw materials. The variables controlling the vapour-phase nitration of ethane have been examined, and data are presented showing the effect of temperature, molecular ratio, contact time, reaction space, and the presence of potassium nitrate on the conversion. Under carefully controlled conditions it is possible to raise the 9% conversion previously reported to 33% based on the nitric acid; the ratio of nitromethane to nitroethane is 27 : 73 by weight. J. W. H.

1179.* **Pressure Gradient Fractionation of Gases.** J. W. Croely and G. C. Le Compte. *Industr. Engng Chem.*, 1940, **32**, 3, 430.—Assuming Henry's Law, equations are developed to show the distribution of a multi-component gas between a solvent and a gaseous phase. The method of using these to calculate the composition of the gas phase obtained as the pressure on a solution is gradually reduced is illustrated by an example. The equations are then extended to calculate the separation obtained in a counter-current system in which solvent flows counter current to gas, the pressure on the solvent increasing towards the top of the column. A means of accomplishing this practically is indicated. P. D.

1180.* **The System Benzene-Nitrogen. Liquid-Vapour Phase Equilibrium at Elevated Pressures.** P. Millor and B. F. Dodge. *Industr. Engng Chem.*, 1940, **32**, 3, 434.—

The vapour-liquid equilibrium was determined at pressures up to 300 atm. and at temperatures from 30° to 125° C. The results are given in tabular and graphical form, and the applicability of Henry's Law is discussed. P. D.

1181.* Detergents from Kerosine. A. R. Padgett and E. F. Degering. *Industr. Engng Chem.*, 1940, **32**, 486.—A fraction boiling at 95–100° C. (15 mm.) was prepared from kerosine supplied by the Standard Oil Co. (Indiana). This fraction, which corresponds to the boiling range of dodecano, was chlorinated to yield about 85% of the monochlorides (kerosyl chloride) which were subsequently converted into *NN*-diethanolkerosylamino by digesting with diethanolamino for 18 hr. at 185° C.

Laurylamino, *NN*-diethyl-laurylamino, and *NN*-diethanollaurylamino, and the corresponding chlorides were prepared for use as reference compounds from lauryl bromide.

The amine hydrochlorides were then compared against sodium lauryl sulphate as a standard, by means of a foam test and an interfacial tension test. The results obtained indicated that the amine hydrochlorides which were prepared were less satisfactory as foaming agents than sodium lauryl sulphate, although the primary amine hydrochlorides appear to be more satisfactory than the secondary compounds. The least satisfactory of the compounds examined as foaming agents were the amine hydrochlorides prepared from the kerosine. H. E. T.

1182.* Viscosity of Hydrocarbon Solutions. Methane-Ethane-Crystal Oil System. B. H. Sage and W. N. Lacey. *Industr. Engng Chem.*, 1940, **32**, 4, 587.—The viscosities of a series of mixtures of the three components were measured at 100° F. and at pressures up to 2500 lb. per sq. in. The majority of determinations were in the condensed region, but some figures were obtained for the liquid phase in the 2-phase region. The per cent. by weight of methane was varied from 0 to 4.7 and of ethane from 1 to 10 approximately. P. D.

1183.* Calculation of Boiling Points of Aliphatic Hydrocarbons. C. R. Kinney. *Industr. Engng Chem.*, 1940, **32**, 559.—The calculation of the boiling points of paraffins, clofines, acetylenes, and naphthenes from the hydrocarbon structure may be carried out by the use of a cube-root function of the molecular boiling-point number. This number is arrived at by adding together the values of the boiling-point numbers assigned to the various atoms, radicles, and linkages. By this means it is possible to calculate the boiling point of hydrocarbons with an accuracy of 5% in most cases. Alternatively, the boiling point and the molecular formula may be used to assist the determination of the hydrocarbon structure. A list of hydrocarbons, the determined boiling point of which differs by more than 10% from the value calculated from the molecular boiling-point number, is given. From an examination of this data it is apparent that certain of the boiling points or structures assigned to the hydrocarbons in this list are in error. J. W. H.

1184. Pressure and Temperature Effects on the Viscosity of Liquids. D. Frisch, H. Eyring, and J. F. Kincaid. *J. appl. Phys.*, 1940, **11**, 1, 75.—A theory of viscosity is advanced based on the energy of activation of the process of pairs of molecules lying in adjoining layers in the liquid rolling over each other. The theory is applied to *n*-pentane, ether, benzene, *isopentane*, water, and mercury, and it is shown that the effect of pressure on liquid viscosity can be calculated with reasonable success from thermodynamic data alone. P. D.

1185.* Analyses Present Evidence on Origin of Petroleum. W. V. Howard. *Oil Gas J.*, 11.4.40, **38** (48), 46.—In an investigation of the relationship of stratigraphy to the type of crude oil, a method of classifying crude oils according to their chemical nature has been worked out. By plotting the reciprocals of the boiling points of 25° boiling range fractions as abscissæ against the specific gravity of the fraction as ordinates it was found that the points representing straight-chain paraffins lie on a straight line, and that no other hydrocarbons fall to the left of that line. It is accordingly given a zero index number. A parallel line through the point represented by benzene was

given a value of 100, practically all other hydrocarbons falling between these limits. *Iso*-compounds have ranges from 5 to 20, the naphthenes between 25 and 50, and the aromatics between 50 and 100. The addition of chains such as pentyl groups to the aromatics or naphthenes lowers their indices so that the index gives an idea of the paraffinicity or naphthenicity of the oil. An examination of a variety of crude oils along these lines has been carried out, enabling the common source of oils from different fields to be correlated and variations in oils in adjacent districts to be distinguished. The work should provide much information of theoretical value concerning the origin, evolution, and migration of crude. For example, where there are families of closely associated oils occurring on definite trends, the existence of two oils lying on a line parallel to this trend, and which are similar to one another and different from those in the trend, may point to the existence of a parallel series of pools. Variations in the oil within a pool may lead to the determination of water levels, if it is found that there is a tendency to increasing naphthenicity or paraffinicity resulting from reactions between the oil and edge water. Sharp variations within pools show the presence of impermeable sections preventing migration and intermixing. This in turn will throw light on well spacing, repressuring, or flooding problems. Much further data on oils of definitely known stratigraphic location is required before any conclusions can be reached as to the origin or migration of oils, in particular the effect of metallic constituents and of sulphur will require studying.

C. L. G.

1186. Boiling Points of *n*-Heptane and 2 : 2 : 4-Trimethylpentane over the Range 100 to 1500-Millimeter Pressure. E. R. Smith. *Bur. Stand. J. Res. (Wash.)*, 1940, 24 (3), 229-234.—In the comparative dynamic method of measuring the b.pt. of liquids, successive measurements are made of the b.pt. of the substance under investigation and the b.pt. of water in ebullimeters connected to the same manostat by means of which the pressure can be varied.

Two simple barometric ebullimeters of the Swietoslowski type were sealed to a manostat through drying tubes.

A diagram and full description of the apparatus and method of procedure are included in the paper.

Data have been obtained from which the following equations were developed to express the relationship between temperature and vapour pressure from 100 to 1500 mm.

For *n*-heptane

$$\log_{10} p = 6.905113 - \frac{1269.821}{217.110 + t}$$

For 2 : 2 : 4-trimethylpentane

$$\log_{10} p = 6.820137 - \frac{1262.707}{221.307 + t}$$

In these equations p is the vapour pressure in standard mm. of Hg exerted by the substance at the temperature t in °C.

D. L. S.

1186a. Separation of the Aromatic Hydrocarbons and the Isolation of *n*-Dodecane, Naphthalene, 1-Methylnaphthalene and 2-Methylnaphthalene from the Kerosine Fraction of Petroleum. B. J. Mair and A. J. Stroiff. *Bur. Stand. J. Res. (Wash.)*, 1940, 24 (4), 395-414.—The kerosine stock used in this investigation came from an Oklahoma crude already used for the investigation of the gasoline and lubricant fraction.

That part of the petroleum distilling 114-144° C. at a pressure of 56 mm. Hg (corresponding approximately to 200-230° C. at 760 mm. Hg) was separated into a series of substantially constant-boiling fractions. The greater part of the *n*-dodecane was then removed by crystallization from the fractions distilling 127-133° C. The remainder of the distillation fractions and the mother-liquors from the crystallization of the dodecane were separated by extraction with methyl cyanide and Marcol into (1) fractions of aromatic hydrocarbons and (2) raffinate fractions containing paraffins and naphthenes with about 2½% aromatics. The aromatics were removed from (2) by adsorption with silica gel.

The aromatic fractions were then systematically distilled in high-efficiency columns and from the material boiling at 56 mm. Hg between 127-127.5° C., 145-146.5, and

148.5–149° C., respectively, naphthalene, 2-methyl-naphthalene and 1-methylnaphthalene were isolated by fractional crystallisation.

“Best” lots of each of the four hydrocarbons were prepared with impurities estimated to be, in terms of mole fraction: *n*-dodecane 0.0000 ± 0.0006 ; naphthalene 0.0006 ± 0.0004 ; 1-methylnaphthalene 0.0025 ± 0.0011 ; 2-methylnaphthalene 0.0000 ± 0.0003 .

The following properties of these hydrocarbons all extrapolated to material of 100% were determined: B.pt. at 760 mm. Hg, f.pt. in air, density, refractive index and refractive dispersion.

D. L. S.

Motor Fuels.

1187. Processes in the Production of Aviation Gasoline. C. Biroreau. *Science et Ind.*, 1940, No. 290 bis (Supplement to *Revue Mécanique*, 26–27).—A general brief review of the processes adopted for the production of a high-performance motor fuel. The development of the petroleum industry towards a chemical industry is stressed.

T. C. G. T.

1188. High-Octane-Number Motor Spirits by Hydrogenation. Anon. *Sci. et Ind.*, 1940, No. 290 bis (Supplement to *Revue Mécanique*), 28–31.—Hydrogenated spirits are being produced in two main groups: (1) spirits of about 76 octane value by the hydrogenation of coal and crude oil and (2) spirits of about 100 octane value by the hydrogenation of polymers—*e.g.*, diisobutylene to *iso*-octane. The established processes for the production of such fuels are reviewed and some plant designs are presented. The tests of typical charging stocks and the yields results from their hydrogenation are tabulated. Some attention is paid to the lead susceptibility of the fuels. Specifications for Army 102 and 104 octane-value aviation fuels are tabulated. A short note on hydrogen production is included.

T. C. G. T.

1189. Catalytic Reforming. Anon. *Sci. et Ind.*, 1940, No. 290 bis (Supplement to *Revue Mécanique*), 35–36.—The value of catalytic reforming in raising octane values of gasolines is demonstrated by a table of the properties of ten gasolines before and after reforming. In one case 99% of a 45 octane-value Mid-Continent gasoline was converted into 53 octane value, whilst in another case 93% of a South Texas gasoline with an octane value of 29 (boiling range 44–209° C.) was converted into 50½ octane value. The catalysts are often natural earths, and 1-tonne will treat 5000 brls.—that is, assuming ten regenerations of the catalyst before it is finally rejected.

T. C. G. T.

1190. New Synthetic Processes for the Production of Motor Spirits. E. Vellinger. *Sci. et Ind.*, 1940, No. 290 bis (Supplement to *Revue Mécanique*, 37–43).—A brief review of the development of alkylation (*i.e.*, condensation of isoparaffins and olefines in the presence of sulphuric acid) is followed by a general survey of the processes involved. Each factor influencing the reaction—*e.g.*, isoparaffin : olefine ratio; time of contact; strength of acid, temperature, acid : hydrocarbon ratio, etc.—is assessed. Data are presented on the tests (including lead susceptibility) of charging stocks and of the resultant products. One interesting table gives the octane values of various fractions of a diisobutylene-*isobutane* alkylation product. According to this table the initial cut 50–65° C. had a knock rating of 92.2, the 98–105° C. fraction had the highest rating—namely, 97.5—and the lowest rating, 79.7, was recorded by the 130–145° C. fraction. The last few fractions show increasing ratings up to 87.3 for the final cut at 185–190° C. Alkylation plants constructed, under construction, and projected are tabulated. Among those under construction or projected, the largest is that of the Magnolia Petroleum Co. for Beaumont, Texas, which will have a daily production of alkylated aviation fuel amounting to 340 m.³. Of the others projected, or being constructed, the most important are three of 240 m.³ per day production—namely, at Baytown for the Humble Oil Co. and at Houston and Wood River for the Shell Co. The Standard Oil Co. are constructing one of 190 m.³ per day at Baton Rouge. The development of alkylation calls for increasing quantities of olefines, and

there has thus been a concurrent development of processes for the dehydrogenation of lower paraffin hydrocarbons to meet this demand. The dehydrogenation processes are outlined.

T. C. G. T.

1191. Anti-Detonants in the Manufacture of Aviation Fuels. H. Guinot. *Sci. et Ind.*, 1940, No. 290 bis (Supplement to *Revue Mécanique*, 45-52).—The increasing demand for high-performance aviation fuels is emphasized by a table showing the percentages of various fuels consumed by the U.S. Army over the years 1932-1938. In 1932 they used 86% of 87-octane-value fuel and 14% with a 58-65 rating. In 1938 90% of their consumption was 100 octane fuel, and this amounted to about 700,000 hectolitres. The demand is increasing at an enormous rate, the consumption being probably quadrupled in three years. The efforts being made to meet demand are reviewed. Dopes such as lead tetraethyl and iron carbonyl, and synthetic fuels, such as alkylated and polymerized hydrocarbons, isopropyl ether, alcohols, ketones, and esters all receive attention.

T. C. G. T.

1192. Patents on Motor Fuel. Standard Oil Development Corp. E.P. 521,786, 30.5.40. Appl. 11.4.39. Fuel for compression ignition engines of the diesel type consisting of a hydrocarbon fuel and a small amount of an organic compound containing in the same molecule a diazo-group and a carboxyl group to impart better ignition qualities to the fuel.

L. A. Mekler. U.S.P. 2,203,833, 11.6.40. Appl. 30.9.32. Improvement in the method of heating light hydrocarbon oils of the character of naphtha and gasoline to effect their pyrolytic conversion. The oil, while substantially in liquid phase, is subjected to heating at a rapid rate, and thereafter at a decreased rate, and substantially completely vaporized during heating at the decreased rate. Afterwards it is again heated at a relatively rapid rate and the oil in vapour phase cracked.

R. Rosen. U.S.P. 2,205,107, 18.6.40. Appl. 23.10.35. Production of a motor fuel containing at least 25% of a polymer blend comprising diisobutylene and hydrogenated diisobutylene, the proportion of the latter being at least three times that of the former.

E. W. Thielo. U.S.P. 2,205,607, 25.6.40. Appl. 26.10.38. Conversion of wax into high-quality motor fuel by vaporizing and heating to a temperature between 850° and 1000° F. The heated vapours are then contacted with a catalyst comprising metal oxides and the reaction products fractionated into at least one gas fraction, a gasoline fraction, and a fraction heavier than gasoline.

H. B. M.

Lubricants and Lubrication.

1193. Friction and Temperature as Criteria for Safe Operation of Journal Bearings. S. A. McKee. *Bur. Stand. J. Res. (Wash.)*, 1940, **24** (5), 491-508.—One of the chief limitations of the science of journal-bearing lubrication in its present status is the difficulty of applying the results of experimental investigations directly to problems involving the performance of bearings in actual service.

This paper describes a method for the application of friction and heat-dissipation data to the determination of the load-carrying capacity of a journal bearing when operating with a lubricant of known characteristics. Two primary considerations are necessary: (1) that the bearing shall always operate in the region of stable lubrication; (2) that the bearing operating temperature shall never exceed some limiting value.

An equation for thermal equilibrium gives the relationship between the thermodynamic and hydrodynamic factors pertaining to journal-bearing lubrication. From this relation, pressure-speed curves representing the limits for safe operation are obtained by consideration of permissible values for temperature rise and the operating

variable $\frac{ZN}{P}$.

An example of the application of this method is given with data obtained experimentally in laboratory apparatus with bearings and lubricant typical of some automotive installations. The method is also used to calculate the effects on the load-carrying capacity of the bearing of changes in the viscosity characteristics of the lubricant, critical $\frac{ZN}{P}$ value, ambient temperature and heat dissipation characteristics.

The practical application of the method is outlined for the case of automotive-engine bearings. D. L. S.

1194.* Fundamental Mechanical Aspects of Boundary Lubrication. H. Blok. *J. Soc. aut. Engrs.*, 1940, **46** (2), 54-68.—The author's compromise classification of boundary lubrication based on two of the mechanical conditions discussed in the paper is distinguished by the following four main types.

- Type I. Mild Boundary Lubrication.
- Type II. High-temperature Boundary Lubrication.
- Type III. High-pressure Boundary Lubrication.
- Type IV. Extreme Boundary Lubrication.

Each of these types is discussed, and examples are given of occurrences in practice with reference to lubrication phenomena and processes and also to improvements to the lubricant and rubbing surfaces.

Thus Type I occurs under conditions of low pressure over a "dispersed" contact area, low temperature, and low sliding speed. Examples are low-speed sleeve bearings, and leaf springs. Greases are suitable lubricants for these conditions, and improvements in oils can be effected by the addition of polar agents.

Type II is subdivided. Thus Type IIA is characterized by high bulk temperature such as occurs in internal-combustion engines. Pressure is low and motion is of a sliding nature. Piston-ring lubrication is an example of this type. Improvement in the lubricant is difficult, inasmuch as the added dope must withstand high temperature, and the property of controlling wear may have to give place to subsidiary properties such as anti-ring sticking and stability.

Type IIB is characterized by high-temperature flashes on the surfaces of actual contact which are of a "dispersed" nature. Sliding speeds must be relatively high. The example given is that of fast-running sleeve bearings where there is actual contact between journal and bearing. Such conditions need a copious oil supply to reduce frictional heat and so minimize the danger of seizure. The lubricant for these conditions is improved by the addition of polar molecules.

Type III, High-pressure Boundary Lubrication, is not to be confused with what is commonly termed at present Extreme-pressure Lubrication, now classified as Type IV. Both types are characterized by "concentrated" contact. In Type III the mean temperature of the region of contact is by definition low. Examples are primarily of rolling contact, as in anti-friction bearings, and also spur gears where the actual contact is on the pitch circle. Oil is the choice for lubricant, since it will flush away worn particles, and so help to keep the surfaces true—a prime essential for this type of contact. Typical forms of wear are pitting and flaking, which may start below the surface as a result of over-stressing of the rubbing materials. In endeavouring to combat both the mechanical and chemical (oxidizing and nitriding) wear no improvement of the lubricant is possible.

Type IV is characterized by "concentrated" contact and high temperature, temperature flashes being especially characteristic. Highly loaded hypoid gears are a classical example. Prevention of seizure is a primary aim, and, as regards seizure, it is temperature and not pressure which is decisive. Design of gears and choice of materials according to their thermal properties will aid in reducing temperature flashes. The lubricant can be improved by the addition of dopes which are chemically active at the high local temperatures. The dopes should not be active at the bulk temperature of the oil in order that undue corrosion be prevented.

The various types of contact are analyzed and defined in an introduction to the paper, and an appendix deals with frictional vibrations under conditions of boundary lubrication. J. L. T.

1195. Studies in Lubrication. VI. Electrolytic Models of Full Journal Bearings. F. Morgan, M. Muskat, and D. W. Reed. *J. appl. Phys.*, 1940, **11**, 2, 141.—The theory, construction, and operation of an electrolytic model of a full journal bearing of journal displacement to radial clearance of 0.1 are described. The results are compared with those calculated from hydrodynamic theory, and agreement is obtained. The model is adapted to the case of bearings with sources of lubricant and circumferential grooves.
P. D.

1196.* Historical Development of our Knowledge of Friction and Lubrication. G. Vogelpohl. *Oel u. Kohle*, 1940, **36**, 89–93, 129–134.—The development of theories of friction from the time of Leonardo da Vinci to the latter part of the nineteenth century is described. Numerous references are made to the original literature, and a table is given of the salient points of the theories of twenty-five authors. In the case of twelve of the more important, more detailed information is made available and illustrations are given of apparatus and diagrams from their publications. Reference is also made to work done on the physical nature of friction and to the development of mathematical theories.
T. T. D.

1197.* Contribution to the Theory of Lubrication of Metal Surfaces at High Temperatures. D. Biro. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 19–26.—This paper deals with the consumption of lubricating oil in aircraft engines. As is well known, the consumption of oil increases with the number of revolutions, and follows an exponential law. The question is treated mathematically, and includes a description of experimental work carried out in this connection.
H. I. L.

1198. Patents on Lubricants and Lubrication. Standard Oil Development Co. E.P. 521,776, 30.5.40. Appl. 28.11.38. Improvement in production of addition agents for lubricating oils. The initial condensation product obtained by condensing halogenated aliphatic hydrocarbons or olefins with aromatic hydrocarbons is further condensed with a dicarboxylic acid halide.

E.I. Du Pont de Nemours & Co. E.P. 522,122, 10.6.40. Appl. 5.12.38. Preparation of lubricating materials having incorporated therein small proportions of entirely organic esters of branched chain aliphatic alcohols containing from six to nine carbon atoms with phosphorus or thiophosphorus acids.

E.I. Du Pont de Nemours & Co. E.P. 522,123, 10.6.40. Appl. 5.12.38. Preparation of lubricants having incorporated therein a small proportion of an alkyl thiophosphite containing at least one alkyl radical directly attached to a sulphur atom of the thiophosphite radical and containing one to six carbon atoms.

Standard Oil Development Corp. E.P. 522,189, 11.6.40. Appl. 2.5.39. Preparation of a lubricating composition containing a mineral oil and a soluble organic xanthogen tetrasulphide.

United States Rubber Co. E.P. 522,191, 11.6.40. Appl. 14.6.39. Improved lubricating oil or grease having dispersed therein a bis-alkyl xanthogene having at least three carbon atoms in the alkyl group and having a decomposition point of at least 150° C. whereby to increase the film strength of the oil at working temperatures.

B. H. Lincoln. U.S.P. 2,204,538, 11.6.40. Appl. 18.3.35. Preparation of a lubricant comprising a major proportion of a hydrocarbon oil and a minor proportion of the sulphurized esters obtained by esterifying with a monohydric or dihydric alcohol the fatty acids obtained from processing pine wood, and then sulphurizing these esters.

L. A. Mikeska. U.S.P. 2,204,609, 18.6.40. Appl. 26.9.36. Improving a naphthenic lubricating oil by the addition of a relatively small amount of an acyl halide. The resulting mixture is condensed in the presence of a catalyst of the Friedel-Crafts type.

C. F. Prutton and A. K. Smith. U.S.P. 2,204,620, 18.6.40. Appl. 26.7.34. Preparation of a lubricating composition containing a major proportion of mineral oil and a minor proportion of both a halogenated organic compound and a sulphurized mineral oil.

R. C. Cantelo. U.S.P. 2,204,660, 18.6.40. Appl. 24.1.38. Preparation of an improved lubricating oil containing a small proportion of thiobenzophenone.

J. M. Musselman and H. P. Lankelma. U.S.P. 2,205,172, 18.6.40. Appl. 26.11.37. Preparation of a lubricant containing a mineral lubricating oil and a small amount of a salt of an *N*-substituted-*p*-aminophenol and an organic carboxylic acid of at least six carbon atoms.

J. A. Wunsch and C. A. Marlies. U.S.P. 2,205,740, 25.6.40. Appl. 14.3.32. Preparation of a lubricant containing a major proportion of unchlorinated mineral oil and a smaller amount of a compound including a chlorinated fatty acid radical.

L. A. Mikeska. U.S.P. 2,205,858, 25.6.40. Appl. 23.1.32. Extreme-pressure lubricant composed of a mineral lubricating oil and a small quantity of polysulphides derived from mercaptans obtained in the refining of petroleum. H. B. M.

Gas, Diesel and Fuel Oils.

1199.* *Relation between Engine Tests and Startability of Diesel Fuels.* P. Weber. *Oel u. Kohle*, 1940, 36, 78-89.—Since it is desirable to be able to rate the efficacy of diesel fuels in starting from cold, an investigation has been carried out on fourteen fuels to find what relationship there is between this property and cetane number as determined on the German War Office test engine. Three engines were employed, including both direct and indirect injection, and for the purpose of the test only one cylinder in each engine was used. The fuels were compared by the throttling method; at constant air temperature the air intake was throttled until ignition ceased to occur. Comparisons were effected by measuring the decrease of pressure in the intake line. Great care was exercised in keeping all working conditions constant, the temperature of the intake air being controlled by running the engines in a refrigerator.

The test engine was driven by an electric motor at 120 revs./min., and after 15 sec. fuel was injected for ten complete cycles, thereafter alternating three cycles with, and three cycles without, injection. From the indicator diagram and revolution counter the time of first ignition was observed. The process was repeated at various temperatures and the time to first ignition plotted against temperature. From the best curve through the points the temperature at which the engine started after thirty cycles was read off and taken as starting temperature. A number of these diagrams are given, showing the deviation from the mean to be expected; all were of similar shape, approaching the time axis asymptotically as the temperature decreased. Graphs are also shown of the relation of starting temperature to cetane number measured on the test engine; these are hyperbolic, and equations are given connecting starting temperature with cetane number. The departures from the curve were few, and the test mixtures of cetane and α -methyl-naphthalene were also found to lie on this curve.

Tests were also run which showed that cetane number dopes such as amyl nitrate had not correspondingly good effects on the starting temperatures, so that the relationship established between the starting temperature and cetane numbers of diesel fuels does not hold if the latter are doped. T. T. D.

1200.* *Influence of Dopes, Particularly Organic Peroxides, on the Ignition Qualities of Diesel Fuels.* A. W. Schmidt and F. Mohry. *Oel u. Kohle*, 1940, 36, 122-128.—High-grade diesel oils must be essentially paraffinic, hence, if aromatic distillates are to be used in C.I. engines, they must either be subjected to drastic refining, with consequent wastage, or treated in some other way to improve their cetane rating. Work on gasoline engines has shown the connection between the presence of organic peroxides and knock, so the effect of peroxides as cetane number dopes has been investigated. The authors divide organic peroxides into four groups: (1) H-O-O-H; (2) H-O-O-R; (3) R-O-O-R; (4) $\left[R \begin{array}{c} \diagup \text{OO} \diagdown \\ \diagdown \end{array} \right]_x$; and investigations were carried out with representatives of all four.

Engine tests were run on a single-cylinder diesel motor with variable compression,

and the variation of ignition delay with compression ratio was determined. Five series of experiments were run: (1) 2% solution of dopes in a diesel oil derived from lignite; (2) 1% solution of dopes in the same basic fuel; (3) 2% solution of dopes in another lignite diesel oil tested after storing for 10 months; (4) 2% and 1% solutions of dopes in a diesel oil derived from coal; (5) 2% solution of dopes in a commercial gas oil. A high-grade Iranian gas oil was also examined in each series as a control.

These series of experiments grade the peroxides in approximately the same order of efficiency; the dialkyl peroxides are most effective, followed by diacetone diperoxide; tetralin peroxide proved the least efficient. As an example of the efficacy of these dopes it may be quoted that the diesel oil derived from coal would not fire satisfactorily with a compression ratio less than 18:1, whereas on the addition of 2% diethyl peroxide similar behaviour was obtained with a ratio of 13:1. T. T. D.

1201.* Relation of Diesel Fuel Properties to their Engine Performance. W. S. Mount and G. A. Hope. *J. Soc. aut. Engrs*, 1940, **46** (3), 100-105, 136.—This paper represents a study of some of the fundamentals which might point to effective means of improving the performance of existing high-speed diesel engines. Only those problems which lend themselves to ready solution in the laboratory were attempted, and tests dealing with combustion-chamber deposits, gum formation, crankcase contamination, engine wear, and similar long-time investigations have been omitted from the discussion.

Seven high-speed engines were used in the work, ranging in size from 3 to 75 h.p. per cylinder, in order to cover a suitable cross-section of the types considered most critical from a fuel-requirement standpoint. Extensive data are given which show the effect of fuel characteristics on combustion and general engine performance, the effect of load and speed on sensitivity to fuel cetane number; and the influence of jacket temperature and injection timing on performance and cetane number requirement, in several types of engine.

It is concluded that although slight improvements in engine roughness are possible with high-quality fuels, the gain is small compared with that which might be obtained by minor design changes. Partial control of variations in ignition delay and combustion pressures caused by varying speed and load can be achieved by the use of higher than average quality fuels, but the control so obtained does not warrant the extra cost of such fuels. Test data indicate that a high combustion-chamber temperature should be maintained and operation at light loads should be carried out at jacket temperatures of 160° F. or higher. Every move towards higher combustion-chamber temperatures seems to improve the combustion process, and if this trend continues without great change in the injection process, it is predicted that very high-quality fuels as determined by cetane number and physical characteristics should take a less important place in future diesel fuel sales. C. H. S.

Asphalt and Bitumen.

1202.* Note on the Measurement of Flow Properties of Asphalts by the Penetrometer. C. C. Mill and V. G. W. Harrison. *J. Soc. chem. Ind.*, 1940, **59**, 66-67.—These authors state that the reasoning in the recent paper by C. Mack (*J. Soc. chem. Ind.*, 1939, **58**, 306) is mathematically unsound, and that the penetrometer equations are more complicated than would appear from his paper. They point out that if two asphalts have the same depth of penetration after a given time, it does not follow that their viscosities are the same, and from this show that Mack's equations are incorrect and their treatment not self-consistent, and hence the viscosity figures given appear unreliable. They also indicate that yield values cannot be obtained by Mack's method, but it is necessary to plot the rate of shear against shearing stress and find the load at which the rate of shear vanishes by extrapolation. H. G. W.

1203.* An Index of the Chemical Nature of Road Tars. T. H. Blakeley. *J. Soc. chem. Ind.*, 1940, **59**, 87-88.—An increase in the severity of the cracking of coal produces a tar which is more aromatic and has a higher specific gravity (s.g.). The s.g., however, depends on the viscosity, and both are necessary to define the tar. The

author derives values called "standard gravities" by giving the s.g. which, according to a graph of equiviscous temperatures (E.V.T.) *v.* s.g. for various tars, would be obtained at a fixed E.V.T. (30° is suggested). On this basis all coke-oven tars would have a value of 1.20 approximately. A table of conversion is given, together with suggested limiting values.

H. G. W.

1204.* **Study of Sieves for Coarse Aggregates.** A. H. D. Markwick. *J. Soc. chem. Ind.*, 1940, 59, 88-92.—Since it had been pointed out that there appeared to be discrepancies between woven wire and perforated plate sieves from $\frac{1}{8}$ to $\frac{3}{8}$ in., comparative tests were carried out, and it was found that the woven wire sieves were considerably less accurate in aperture size and shape. Owing to the use of crimped warped wires in manufacture, the openings were found to be trapezoidal with alternate short and long sides, and owing to the interweaving of the wires it was shown that particles of a larger size than the projected horizontal size could be passed through. The effective size was found to be 3-4% greater than that of a corresponding perforated plate. To offset wear during tests a very hard igneous rock with its edges rounded off was used, and tests of 5 min. duration were made on the woven and perforated sieves alternately. By means of sieving tests on hand-gauged samples of $\frac{3}{8}$ -in. size the effective sizes of the two types of sieve were obtained. The differences between the effective size and the average projected aperture were then accounted for by correcting for the effect of the woven aperture and by making an allowance for the variability in aperture size. The rate of passage through both sieves was found to be practically the same. Flaky material was found to pass more slowly than cubic, and elongated material was very difficult to sieve. To avoid two discontinuities in the sieve series it is suggested that perforated plates only be used down to $\frac{3}{16}$ in., where there is already a break in the sieve series, otherwise, if the change is made from perforated plate to woven wire before this, serious errors may arise in certain cases.

H. G. W.

1205.* **Research in Soil Stabilization with Emulsified Asphalt.** C. L. McKesson. *Proc. Amer. Soc. Testing Mats.*, 1939, 39, 1123-1133.—Soil stabilization with emulsified bitumen has come into extensive use only during the last five years. Prior to this some research studies had shown that the amount of emulsion required varied widely for different soils and depended on the consistency of the bitumen and type of emulsion. The development of test methods to measure the efficiency of stabilization was therefore necessary, since the usual methods of testing hydrophilic soil were unsatisfactory with the hydrophobic bitumen-covered soil. Two of the test methods, the absorption test and the test for stability, have been recommended as tentative standards. The present absorption test was developed from the simple soaking of stabilized samples in water and is described in detail, parallel tests being run on the stabilized and unstabilized soils. Compacted cylinders of 2 by 4 in. dried to constant weight at 140° F. are placed on heavy water-saturated cotton-flannel cloth and covered to prevent evaporation. Seven days was found to be sufficient to decide whether stabilization was effective or not, depending on the amount of absorbed water. The bottom $\frac{1}{2}$ in. is used in the stability test and is fully saturated after this time. A properly stabilized specimen absorbs only 10-15% of the water absorbed by an unstabilized one, and the limit decided on was 12% of that in the untreated specimen plus 0.5%. For the stability test the compacted cylinders, after testing as above, are placed in the stability test apparatus and the saturated lower part extruded $\frac{1}{2}$ in. through a circular orifice of 1 sq. in. The total load in lb. required is taken as the stability. These two tests have been universally specified since 1935 and have given every indication of the efficiency of a stabilization project. The height of the specimen does not appear to be critical, neither does wall friction. Loads up to 40,000 lb. are required and a minimum of 10,000 lb. is considered satisfactory. The plastic flow was found to be greatest in the centre of the specimen. A punching shear test is also described, which, it is thought, may bring out additional valuable information. The test methods are used to indicate the relative efficiencies of different types of emulsions and to determine the quantities required. As examples it is shown that a fine emulsion is much more effective than a coarse one and bitumen of 58 pen. 10% more effective than bitumen of 209 pen.

H. G. W.

1206.* **Evaluating the Adhesive Properties of Asphalt.** A. Holmes. *Proc. Amer. Soc. Testing Mats.*, 1939, **39**, 1140-1149.—The "adhesivity" of an asphaltic bitumen is defined as its ability to resist being displaced by water from an aggregate. To evaluate this property the following tests have been used:

(a) The partition test, which measures the amount of non-oil-wetted material after shaking a mixture of fine aggregate, water, and asphaltic oil, is considered to give only very rough results.

(b) Various water-displacement tests, in which the aggregate is coated with bitumen and then subjected to the action of water at a given temperature, appear to give the most promising results. The author uses 20 hrs. soaking in distilled water at room temperature for uncured cutbacks, and at 140° F. for completely cured cutbacks and paving bitumens. A further accelerated test is to boil in distilled water for 1-5 min. The "adhesivity value" is the percentage remaining coated and is estimated visually.

(c) In the abrasion-displacement tests coated mineral aggregate is shaken under water in a can in a shaking machine until the test is completed, and is generally the time required to remove a given percentage—*e.g.*, 50%.

It is considered that test (b) gives the most indicative results, preference being given to that at 140° F., since (c) is subject to the errors of (b) as well as abrasive effects.

To obtain results of a more practical character circular track tests have been developed to a standard procedure, and details of its operation are given. The breakdown of the track is taken as the number of revolutions of the machine required to loosen 10% of the total bitumen-aggregate mixture laid. Between 78° and 90° F. there is a mean increase of 840 revs./degree decrease in temperature and at 90° F. an increase of 450 revs./degree rise in softening point, the track being run under water and continuously in each case. A correlation has been indicated between the results obtained on the circular track and the laboratory tests (b). By the use of suitable wetting agents, particularly heavy metal soaps, in the bitumen the track life has been increased up to 3.88 times that of the untreated bitumen. A considerable amount of work remains to be done to develop a small-scale quantitative laboratory test which can be correlated with road results.

H. G. W.

1207.* **Some Factors Affecting the Thermal Susceptibility of Asphaltic Paving Mixtures.** R. Vokac. *Proc. Amer. Soc. Testing Mats.*, 1939, **39**, 1153-1157.—The compression test was used to measure the strength or stability of asphaltic bitumen paving mixtures because of the correlation obtained with the service performance of mixtures. Values were obtained at various temperatures with various mixtures using a hydraulic Baldwin-Southwark compression tester. Data are given showing the influence of mixture composition on the susceptibility of compressive strength to change in temperature and in asphalt cement; this thermal susceptibility was found to vary with variation in each of these three factors. In comparable mixtures it has been shown that the thermal susceptibility varies according to that of the asphalt cement. Although additional filler decreases the susceptibility of a given mixture, the effect is not so evident at low temperatures, and is also markedly influenced by the source of the asphaltic bitumen. It is therefore concluded that, in order to obtain mixtures having very similar physical characteristics, such as susceptibility, strength, etc., if bitumens from different sources are used, the mixtures must be made up to suit the individual characteristics of each bitumen.

H. G. W.

Special Products.

1208.* **Rubber from Petroleum.** H. S. Norman. *Oil Gas J.*, 11.4.40, **34** (48), 9.—Plans for the commercial production of Buna rubber at Baton Rouge, La., are in the early stages of development, a plant with a daily capacity of 10,000 lb. synthetic rubber being considered. In modern practice it is derived from butadiene combined with acryl nitrilo and styrene, and is claimed to have most of the desirable properties of natural rubber with a superior resistance to heat, abrasion, ageing, and swelling in

gasoline and oil. Butadiene may be produced by the catalytic dehydrogenation of *n*-butane to butylene at 25–80 lb./sq. in. and 900/1000° F., followed by conversion of the butylene at normal or reduced pressure and 1000/1200° F. After removal of the butadiene, the residual butylene may be polymerized, the unpolymerized hydrocarbons separated and re-circulated to the first step, and the butylene depolymerized and re-circulated to the second step. Butadiene is extracted from the liquid hydrocarbons with liquid ammonia or ethylene glycol or absorbed from the gases by acid cuprous chloride.

Butadiene is also derived from propylene or cyclohexane pyrolysis at 450–800° C. under atmospheric or reduced pressure, the pyrolysis products being rapidly cooled to retard further reaction and to condense the hydrocarbons. Butadiene is separated by cooling and compression, by absorption in low cold test oil or solution in *e.g.* mercuric salt solution or liquid SO₂. It is almost invariably formed during pyrolysis of hydrocarbons at lower temperatures than those at which aromatic hydrocarbons are formed. Butadiene-1 : 3 is obtained by the catalytic dehydrogenation of buteno-1 or -2 under controlled conditions, and isoprene from branched-chain pentenes. Once through yields of butadiene from *n*-butylenes varied from 20 to 30%, with ultimate yields of 80%.
C. L. G.

1209.* Distribution of Nicotine between Water and Petroleum Oils. L. B. Norton. *Industr. Engng Chem.*, 1940, **32**, 241.—At the low concentrations used in insecticidal spray mixtures, nicotine is almost equally distributed between the oil and water phases. At intermediate concentrations the distribution is in favour of the water, reaches a maximum, and decreases at high concentrations. The presence of acids leaves any excess of free base equally distributed between the two phases, and when present in excess results in the complete extraction of the nicotine into the aqueous phase. At concentrations of less than 0.1*N* alkalis have no effect on the phase distribution, at higher concentrations they drive the nicotine into the oil phase, the extraction into the oil phase being complete with 5*N* sodium hydroxide. The result of these observations is that although it is highly desirable in insecticidal sprays that the nicotine should be concentrated in the oil phase, it is distributed equally between the oil and water phases independently of nicotine concentration, the presence of fungicides, and emulsifiers. Drying of the spray on the foliage concentrates the nicotine in the oil phase.
J. W. H.

1210.* Plastics and Resins from Hydrocarbons. P. K. Frolich. *Industr. Engng Chem.*, 1940, **32**, 293.—The magnitude and comparative rates of growth of the production of resins from coal tar and non-coal tar sources are reviewed. It is concluded that, although the quantity of non-coal tar resins is only one-eighth of the total synthetic resin production, it is a very rapidly expanding product. A list is given of the hydrocarbons which are used in the production of synthetic resins and rubber. J. W. H.

1211.* Propane Precipitation of Petroleum Resins. P. T. Graff and H. O. Forrest. *Industr. Engng Chem.*, 1940, **32**, 294.—Propane precipitation of petroleum resins is used commercially to reduce the duty of clay in decolorizing lubricating-oil stocks. The de-resinification process follows the conventional propane deasphalting treatment, in which asphalt precipitation takes place with six volumes of propane at 100° F. After removal of the asphalt, a further 200 volumes of propane are added and the temperature is raised to 140° F., when the primary resins are precipitated. These resins are washed free of oil with propane, or may be fractionated into washed resins and high- and low-viscosity stocks by the application of a temperature gradient to the propane washing treatment.
J. W. H.

1212.* Polybutenes. R. M. Thomas, and others. *Industr. Engng Chem.*, 1940, **32**, 299.—The polymerization of butenes at low temperatures yields high-molecular-weight polymers, which vary from viscous liquids to elastic solids. These polymers are chemically inert and stable in character. The technique employed in the determination of the molecular weights of these polymers is described. The addition of polybutene to cellophane is shown to increase the impermeability to water vapour.

The addition of 25% Exanol (a 15,000-molecular-weight polybutene) to a low viscosity and low V.I. lubricating oil, results in the production of an extremely viscous (3180 secs. Saybolt at 210° F.) lubricating oil, possessing a V.I. of 130. Viscosities of 14,000 secs. Saybolt at 210° F. may be obtained by the addition of only 5% of a 73,000-molecular-weight polymer to a low viscosity base stock. These very high-molecular-weight polymers impart a stringy character to the product, which is of value in reducing oil leakage in open-type bearings and in any place where excessive oil must be avoided. By the addition of small amounts of polybutenes, the properties of asphalt may be modified appreciably, the softening point and penetration are increased, and the slope of the penetration-temperature curve lowered. The addition of polybutene to paraffin wax increases the viscosity of the wax, the low-temperature flexibility and the bonding properties.

J. W. H.

1213.* Production of Petroleum Resins. S. C. Fulton and A. H. Gleason. *Industr. Engng Chem.*, 1940, **32**, 304.—Resins can be produced from cracking coil tar by three methods:—

(a) The cracking coil tar is reduced in a high vacuum to produce a residuo having a softening point of 110° C. This residuo is extracted with a refined petroleum distillate (*e.g.* Varsol). The suspension of finely divided asphaltenes is filtered off and the solution of resins treated with sulphuric acid and clay. The resin is finally recovered by distillation of the solvent. The yield of resin on the original tar is approximately 7%.

(b) The tar is distilled to a low-melting-point pitch, which is treated with aluminium chloride. This treatment causes coalescence of the dispersed asphaltenes (which are filtered off), as well as the formation of additional resins. After acid and clay treating the aluminium chloride treated tar, the resins are recovered by distillation under high vacuum. A yield of 11% resin on the reduced tar may be obtained by this method.

(c) The distillate from cracked petroleum tar is chlorinated and the product heated to 350° C. under vacuum to remove the overhead products (hydrochloric acid and oily materials). The residuo is treated with naphtha and the hydrocarbon insoluble material separated from the resins, which are recovered by distillation of the solvent. A resin yield of 15% on the tar distillate is obtained. This process may be modified by the treatment of the chlorinated tar with a condensing agent to increase the yield of resins.

Resins can also be produced by the catalytic polymerization of cuts from light petroleum aromatic extracts with formaldehyde. These resins are pale yellow in colour, brittle, and behave well when used in the preparation of paints and varnishes.

J. W. H.

1214.* Phenolic Compounds from Petroleum Sources. E. Field, F. H. Dempster, and G. E. Tilson. *Industr. Engng Chem.*, 1940, **32**, 489.—A historical summary is presented of the occurrence and nature of phenolic materials, together with their properties, which may be recovered from petroleum sources. Light, medium, and heavy grades of phenolic compounds derived from petroleum have been subjected to careful fractionation and the large number of fractions obtained analysed. The fractions were collected in 3° C. cuts and the physical chemical and germicidal properties of the cuts obtained. The various fractions were finally analysed for individual phenolic compounds according to the neat up-to-date methods in the literature, the analyses being on a quantitative basis. Comparative data were also obtained on a commercial grade of cresylic acid derived from a coal-tar source. The following phenols were identified, *o*-, *m*-, and *p*-cresol, 1:4:2-xyleneol, 1:3:4-xyleneol, 1:2:3-xyleneol, 1:3:5-xyleneol, 1:2:4-xyleneol, *isopseudocum*inol and 1:3:5-methylolthylphenol.

H. E. T.

1215.* Naphthenic Acids from Gulf Coast Petroleum. R. W. Harkness and J. H. Bruun. *Industr. Engng Chem.*, 1940, **32**, 499.—A complex mixture of naphthenic acids has been isolated from Gulf Coast petroleum, by distillation of the lubricating oil fraction over sodium. The naphthenic acids were thus concentrated in the residuo as sodium naphthenates from which they were liberated by acid treatment. The

crude acids were further purified by treatment with alcoholic potassium hydroxide, and the hydrocarbon impurities extracted from the alcoholic solution by means of liquid butane at 70–90° C. in a pressure extractor. The oil free soaps thus obtained were treated with dilute sulphuric acid to liberate the pure mixed naphthonic acids which had a saponification and acid number of 160. The crude acids were separated into three cuts by distillation and each cut further divided into six fractions by distillation in a molecular still. Molecular weights were determined on the fractions by the cryoscopic method in benzene; in addition the saponification number, acid number, bromine number, CO_2H groups per mol., and ultimate analysis were carried out.

From the data accumulated it is concluded that—

1. The specific gravities of the naphthonic acids derived from Gulf Coast petroleum decreased with increase in molecular weight.
2. The higher boiling naphthonic acids are of the monobasic type.
3. The average empirical formulae indicate that the composition ranges from $\text{C}_n\text{H}_{2n-4}\text{O}_2$ up to $\text{C}_n\text{H}_{2n-10}\text{O}_2$, the average number of carbon atoms in the molecule being 14 to 29.

H. E. T.

1216.* Conversion of Naphthonic Acids to Naphthene Hydrocarbons. G. E. Goheen. *Industr. Engng Chem.*, 1940, **32**, 503.—A fraction of naphthonic acids isolated from the lubricating oil portion of a Gulf Coast crude petroleum has been investigated in detail. The isolated naphthonic acids had an average molecular weight of 317 and an average molecular formula of $\text{C}_{20.7}\text{H}_{35.2}\text{O}_{2.1}$. The crude acids were converted by way of ethyl esters, alcohols, and iodides to the corresponding naphthene hydrocarbons, which were found to possess higher viscosity indices than naphthenes from a typical naphthene-base oil.

The crude naphthene hydrocarbons were refined by treatment with concentrated sulphuric acid and a portion was hydrogenated in a methylcyclohexane solution on a Raney nickel catalyst in an Adkins apparatus at a temperature of 230–250° C. and a pressure of 2700–3000 lb. per square inch hydrogen pressure. Physical properties of the products are reported.

After refining with sulphuric acid, the naphthene hydrocarbon had a kinematic viscosity index of 30, a viscosity gravity constant of 0.8604, a gravity index of 55 and kinematic viscosity of 13.41 centistokes at 100° F. and 2.72 centistokes at 210° F.

From considerations of the properties of the naphthene hydrocarbon before and after hydrogenation, it became apparent that the deficiency in hydrogen in the naphthonic acids from which they were derived was to only a small proportion of aromatic material. Evidence obtained indicated that the acids were mainly polycyclic naphthonic acids with an average of 2.6 rings per molecule.

H. E. T.

1217.* Naphthonic Acids (Aruba Acid). J. R. M. Klotz and E. R. Littmann. *Industr. Engng Chem.*, 1940, **32**, 590.—Aruba naphthonic acid is extracted from Colombian gas oil with dilute sodium hydroxide and recovered from the alkaline solution by acidification with sulphuric acid. A typical Aruba naphthonic acid has the following typical analysis:

Sp. Gr. (° A.P. 1)	15.7 (0.961)
Acid Number	202
Saponification Number	208
Iodine Number	4.1
Unsaponified material, %	13.5

A sample of Aruba acid was fractionated and 10% fractions were obtained, with a 20% residuo. The phenol content of each fraction was determined, as well as the effect of each fraction on the air oxidation of benzaldehyde in the presence and absence of manganese naphthenate catalyst. Further, some characteristics of the acids in each fraction were determined.

It has been shown that phenolic compounds occur mainly in the low- and high boiling ends of Aruba acid, and that the intermediate fractions contain only relatively small proportions of phenols. From the experimental results obtained it appears

that the general use of the acid number as a criterion for the purchase or evaluation of naphthenic acids does not give sufficiently detailed information to be of much value to the consumer. H. E. T.

1218. Formaldehyde from Methane. Anon. *Chem. Tr. J.*, 7,6.40, 106, 353.—A claim is made in E.P. 520,080, granted to Gutehoffnungshütte Oberhausen A.-G. for the production of formaldehyde by the partial oxidation of gaseous or vaporous hydrocarbons, of the acetylene, ethylene, or methane series, in the presence of small quantities of nitric or nitrogen oxides and solid catalysts (Si, Zn, Mg, Ti, Ce, etc.) in the form of oxides. A mixture of 49.8% by volume of a gas separated from coke-oven gas (containing 41% of methane), 50% of air, and 0.2% of nitrogen oxides, is passed at a rate of more than 75 cm. per sec. through reaction ducts lined and charged with solid oxide catalyst and heated to 620° C. The reaction gases are circulated several times, the yield being 560 gm. of 100% formaldehyde per cu. metre of methane.

C. L. G.

1219. Patents on Special Products. Isidor Morris Keilbron. E.P. 521,749, 30.5.40. Appl. 25.10.38. Manufacture of new pyridyl phthalic acid derivatives by the interaction of pyridine with a diaotized aminophthalonitrile, aminophthalimide, or aminophthalic dialkyl ester which is in aqueous solution.

May and Baker, Ltd. E.P. 521,821, 31.5.40. Appl. 28.11.38. Preparation of *p*-aminobenzene-sulphonamido-derivatives of glyoxaline by the condensation of an amino-derivative of a glyoxaline with a benzene-sulphonyl halide or anhydride containing in the *para* position an acyl-amino-group which is then converted into an amino-group by hydrolysis.

Charles Pfizer and Co. E.P. 521,831, 31.5.40. Appl. 28.6.39. Conversion of esters of 2-keto-hexonic acids into the corresponding ascorbic acids by treating esters of the 2-keto-acids in aqueous solution in the presence of a metal selected from the group consisting of magnesium, iron, nickel, cobalt, manganese, cadmium, and zinc.

Karnbolaget Aktiobolag. E.P. 521,944, 5.6.40. Appl. 12.9.38. Manufacture of thio-esters by the addition of a thiocarboxylic acid to an unsaturated organic compound or to a mixture of such compounds.

Deutsche Hydrierwerke A.-G. E.P. 522,033, 6.6.40. Appl. 2.12.38. Manufacture of high-molecular ketones. Halides of aliphatic or cycloaliphatic monobasic carboxylic acids having at least six carbon atoms in the molecule are treated with strong tertiary bases.

J. G. Fife. E.P. 522,151, 11.6.40. Appl. 1.12.38. Preparation of improved textile oils comprising a mixture of a mineral oil and higher aliphatic alcohols or ketones having more than 8 carbon atoms in the molecule and a small proportion of a sulphated or sulphonated compound which reduces the surface tension, e.g. sulphuric acid ester salts containing higher alkyl radicals or alkali sulphonates derived from mineral oil sulphonic acids.

The Distillers Co., Ltd. E.P. 522,234, 12.6.40. Appl. 10.12.38. Conversion of olefines into olefine oxides by direct oxidation with the aid of a catalyst. The catalyst is composed of substantially pure silver in finely divided condition, and is supported on a metal support in intimate admixture with an inert filler or solid diluent material.

N.V. de Bataafsche Petroleum Maatschappij. E.P. 522,450, 18.6.40. Appl. 8.12.38. Improved process for the removal of acid components from hydrocarbons or derivatives thereof by extracting with an aqueous solution of an alkali metal.

J. Winkler. E.P. 522,451, 18.6.40. Appl. 8.12.38. Preparation of hydrocarbons from acid-refining waste by neutralizing by the dry method in an oil solution with exclusion of water. Neutralization is effected under pressure in a closed vessel at a temperature above 100° C., and only solvents rich in unsaturated and aromatic hydrocarbons are used for acid refining the waste.

B. T. Thurman. U.S.P. 2,204,109, 11.6.40. Appl. 6.10.38. Separation of minor constituents including phosphatidic material from vegetable oils containing same without destruction of the phosphatidic material. A small proportion of a solution of hydrogen peroxide is mixed with the oil to precipitate the minor constituents, which are afterwards separated from the oil by difference in specific gravity.

R. L. Humphreys. U.S.P. 2,204,597, 18.6.40. Appl. 23.2.37. Preparation of a compounded mineral oil containing a metal salt of an organic acid in sufficient amount to render the oil corrosive to bearing metals, and an organic ester having an unsaturated carbon to carbon bond conjugated with a carboxyl group of the ester. The organic ester is present in sufficient amount to reduce the corrosivity of the compounded oil.

R. L. Humphreys. U.S.P. 2,204,598, 18.6.40. Appl. 23.2.37. Preparation of a compounded mineral oil containing a metal salt of an organic acid and an organic ester of a polycarboxylic acid having two carboxyl groups no more than two carbon atoms apart in sufficient amount to reduce corrosivity of the compounded oil.

F. W. Kavanagh, V. N. Borsoff, and R. L. Humphreys. U.S.P. 2,204,601, 18.6.40. Appl. 23.2.37. Manufacture of a compounded mineral oil containing an addition agent in sufficient quantity substantially to increase the corrosivity of a mineral oil, and a small amount of an organic hydroxyl ester in an amount sufficient to reduce corrosivity of the oil.

F. R. Moyer. U.S.P. 2,204,967, 18.6.40. Appl. 25.9.33. Method of lowering the pour point of waxy mineral oils by incorporating therewith the active substances separated from pyrogenous condensation products from mineral oil by precipitation with an aliphatic solvent.

F. M. Clark. U.S.P. 2,205,187, 18.6.40. Appl. 31.7.36. Purification of a liquid halogenated hydrocarbon containing unfilterable finely divided carbon by dispersing in it a dilute aqueous solution containing at least 3% by weight of salt producing polyvalent cations.

H. Hopff and H. Schoenherr. U.S.P. 2,205,408, 25.6.40. Appl. 29.5.39. Preparation of fluorescent compound containing a hydrocarbon oil and a small percentage of a fluorescence-exhibiting condensation product obtained by heating an aromatic ketone having at least four condensed rings with an anhydrous halide of a metal selected from the group of zinc, beryllium, and aluminium. H. B. M.

Detonation and Engines.

1220.* **Some New Investigations on Old Combustion-Engine Problems.** G. Eichelberg. Parts I-V. *Engineering*, 148 (3850, 3853, 3855, and 3858), 463-466, 547-550, 603-606, and 682-686, and 149 (3871), 297-399.

Parts I-III. *Heat Flow in Cylinder Walls and Pistons and Temperature Effects.*—The heat flow in cylinder walls and pistons is examined both from the theoretical and experimental aspects. The varying temperatures of the cylinder gases are deduced, and from the given data on the heat transfer to the cooling water, the distribution of temperature is estimated.

The differential equation for periodic heat-flow is given from which the surface temperature fluctuations are calculated. The thermocouples used for the experimental measurements are described and the method is given by which the surface temperature variations can be obtained from the readings of thermocouples which are located a small distance beneath the surface.

From a knowledge of the heat transmission from the gases to the combustion chamber surface, and from the piston to the cylinder liner, the temperature distribution and fluctuation in the piston can be arrived at. The author includes measurements of piston and piston-ring temperatures at various points to produce the diagrams illustrating temperature fields, and data are given for both two- and four-stroke engines. The effect of the heat conductivity of the piston and cylinder materials is included.

The stresses produced by the temperature gradients and cyclic temperature variations are calculated for straightforward cases.

Data are given for exhaust-valve temperatures, and the cooling effect through the seat and stress is examined. Diagrams are given showing the temperature distribution and cyclic variation of temperature at different points of an exhaust valve.

Part IV. (a) *Entropy*; (b) *Fuel Injection*.—The use of the entropy diagram in studying combustion problems is put forward and is illustrated by an examination of a Büchi supercharged engine.

Types of fuel-injection pumps and nozzles are described, together with the advantages and disadvantages of the various systems. Some notes on a system which gave satisfactory results with petrol injection at high speeds are given.

The problem of nozzle carbonisation is also discussed.

Part V. (a) *Piston-ring Problems*; (b) *Practical Applications*.—The problems of blow-by and friction in piston rings are discussed, and the results of experimental investigations are given.

Some practical applications of experimental results in modern engine development are mentioned, and in particular the Pescara free piston generator is cited.

J. G. W.

1221.* *Use of Gas as a Fuel for Motor Vehicles*. J. S. Clarko. *J. Inst. aut. Engrs*, 1939, 8, No. 2, 43-76.—This paper gives details of experiments carried out by the City of Birmingham Gas Committee since 1931. The author first deals with thermodynamic and mechanical problems which arise in compressing town's gas to a pressure of 5000 lb. per square inch, and special items of equipment needed for vehicles running on compressed gas, such as gas cylinders, reducing valves, gas mixers, etc., are dealt with from the point of view of the most satisfactory designs and layout on the vehicle.

For similar engines of the same compression ratio running on petrol and gas fuel the optimum power output on gas is approximately 88% of that obtainable on petrol. Data relating to bench tests made on a 20-h.p. Austin engine show the improvement in power and brake thermal efficiency effected by increasing the compression ratio from 4.6 to 5.6 to 1, and analyses of the exhaust gases show that no carbon monoxide was present.

The loss in power when using gas fuel results in poorer acceleration. Tests made on two 6-cylinder Guy buses, one using petrol and the other running on gas, are described, and it was found that by increasing the compression ratio of the gas-driven engine, acceleration in third gear was nearly equal to that obtained with the petrol vehicle. Gas consumption figures for various vehicles are also given.

Experimental work with a Bolliss & Morcom 4-stroke vertical 2-cylinder engine of 3- $\frac{1}{2}$ in. bore \times 5 in. stroke using gas injection and spark ignition, or compression ignition to determine the maximum compression ratio which could be used before pre-ignition developed showed that (1) gas consumption could be reduced by more than 30% when using a compression ratio of 15:1 as compared with a ratio of 7:1; (2) gas could be used without pre-ignition up to compression pressures of 600 lb. per square inch—i.e., 17:1 compression ratio; (3) more power was developed at 900 r.p.m. on gas than on fuel oil, the outputs being 10.29 h.p. and 8.6 h.p.; (4) it was impossible to obtain self-ignition of coal gas at a compression ratio of 17:1, which eliminated the possibility of running small-bore engines on self-ignition principle.

Appendices deal with the calculation of calorific value of compressed gas and estimated costs of a compressed gas scheme for a fleet of buses.

C. H. S.

1222.* 1939—Bulletin. Diesel Engine Users Association.—The latest annual bulletin of the D.E.U.A. is less comprehensive than usual, owing to inevitable war-time restrictions. Nevertheless, it remains a valuable survey of diesel developments in 1939, in the fields of road and rail traction as well as stationary and marine installations. The general tendency for land power has been to increase B.M.E.P., and pressure-charging is being increasingly used. There is a growing interest in engines capable of running on gas and oil fuel alternatively. For marine purposes the chief types built comprised 4-stroke single-acting supercharged, 2-stroke single- and 2-stroke double-acting. Developments in diesel rail traction have been most marked in the Americas, Denmark, Holland, and Italy. Few, if any, major changes have taken place in road transport.

K. A.

1223.* **Modern Aircraft Valves.** A. T. Colwell. *J. Soc. aut. Engrs*, 1940, 46 (4), 147-165.—This paper is a survey of development and current practice in regard to aircraft-engine valves, both in America and Europe. The ability of the modern valve to withstand arduous conditions is attributed to three major developments: sodium cooling, forging technique, and cylinder-head design. Much information is given concerning materials and methods of manufacture employed by the leading engine-builders of both continents, and the paper is comprehensively illustrated. K. A.

1224.* **Aircraft Spark-Ignition Engines with Fuel Injection.** O. W. Schoey. *J. Soc. aut. Engrs*, 1940, 46 (4), 166-176.—The National Advisory Committee for Aeronautics has conducted a large number of tests on single-cylinder engines using fuel injection and spark ignition; three of these were water-cooled and three were air-cooled. Four types of injection pump were used, of which one was a diaphragm type of N.A.C.A. design. Four designs of injection valve were tested.

It was found that performance is not critically sensitive to injection valve location, start of injection, or injection period; maximum power and best economy are generally obtained when the fuel-injection period occupies that part of the cycle preceding the point at which combustion air is entering the cylinder at maximum rate. The fuel spray should be directed in a horizontal plane against the incoming air. Varying the injection-valve opening pressures from 300 to 3000 lb./sq. in. gave no measurable change in engine performance.

Comparative tests of carburettor, cylinder injection and manifold injection showed that m.e.p. closely follows the volumetric efficiency, which is greatest at high speeds when the cylinder-injection method is used. Minimum specific fuel consumption was the same with each method of mixing the fuel and air. By using a valve overlap of 120-130°, a four-valve cylinder can be completely scavenged with 3 to 5 in. Hg pressure difference when fuel injection is employed.

Tests were made using "safety" fuels of volatilities ranging from 205-330° F. to 400-635° F. It was shown that increasing the distillation temperature has only a negligible effect on performance up to the range 350-500° F. The distillation temperature at which performance starts to fall off is approximately the same as the cylinder head temperature. Starting a cold engine with safety fuel is difficult, unless gasoline is supplied to the priming system. Another method which proved successful was the use of propane. K. A.

Coal and Shale.

1225. **Low-temperature Carbonization: A Novel American Method.** Anon. *Chem. Tr. J.*, 24.5.40, 106, 330.—The *Colliery Guardian* for 12th May gives an account of a paper to the American Institute of Mining and Metallurgical Engineers by C. E. Leshner describing the Disco process for the production of low-temperature coke in ball form from coal dust (below $\frac{3}{8}$ in. size). A plant is in operation at Pittsburgh of capacity 7000 tons per month of low-temperature coke and 140,000 gals. of tar. The raw coal is first partly oxidized, to reduce the agglutinating properties, for about 2 hr. in horizontal cast-iron hearths, the coal being stirred and conveyed by rabble arms. Coal is transferred from the roaster at 600° to a reservoir, where it is kept hot by heating gas in ducts and mixed before transfer to the carbonizers. These consist of inclined rotating steel cylinders surrounded by an insulated stationary shell. Heating gases are passed through the annular space. Into the upper end of the revolving rotor are introduced coal and a definite proportion of below $\frac{1}{4}$ in. proheated coke breeze, the low-temperature coke being discharged from the lower end in the form of balls. Cooling of coke balls below 2 in. diameter is effected by water, but smaller sizes are cooled in air, as water-cooling causes shrinkage cracks which destroy the structure. The Disco coke is dense grained and black, consisting of irregularly rounded balls or fragments of balls. The shatter index is above 70% retained on a 2-in. screen. C. L. G.

1226. **Patent on Coal and Shale.** George William Johnson. E.P. 522,096, 10.6.40. Appl. 10.3.38. Process for the pressure extraction of coals which comprises the

employment as an extraction agent of an oil fraction of a medium boiling range, containing not more than 10 gm. of hydrogen for 100 gm. of carbon, together with less than 20% of an oil of a similar boiling point containing at least 14 gm. of hydrogen for 100 gm. of carbon.

H. B. M.

Economics and Statistics.

1227.* **Rumanian Petroleum Exports during the First Three Months of 1940.** Anon. *Monit. Pétr. roum.*, 1940, 41, 605-608.—In the first three months of 1940 Rumania exported 823,507 tonnes of petroleum products, against a figure of 1,130,757 in 1939. These exports are analysed and tabulated according to the country and destination, their value, and the balance of trade with the countries concerned.

The most notable feature of the analysis is that England has increased her percentage takings of the oil exported—i.e., 19.57% to 38.49% or from 221,246 tonnes to 316,996. Germany and Italy, however, have both taken less. Thus, Germany received in the period under review 75,254 tonnes, or 9.14%, instead of 182,790, or 16.17%, in 1939. Italy received 83,436 tonnes, or 10.13%, instead of 172,352, or 15.24%, in 1939.

France received 4.24% of the exports instead of 5.24% in 1939, but Turkey shows a very big increase, from 0.73% in 1939 to 7.05% this year.

Of the 823,507 tonnes of petroleum export reviewed, 367,111 were gasoline, 139,919 kerosine and white spirit, 140,115 gas oil, and 132,296 fuel oil.

The article also tabulates the individual companies supplying the export demands.

T. C. G. T.

1228.* **National Supply of Fuels.** Anon. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 15-18.—A recent publication of statistics by the Institute of the Study of Transport Economics containing figures referring to consumption, import, and national production of fuel, including coal, petroleum, and alcohol, appears to have led to erroneous conclusions for which there is no justification. This article deals fairly fully with the position as it is affected by the present war, and compares the present situation with that which obtained in 1914. It is shown that in the latter year the total consumption of fuel was 2,901,603 tons, of which 84.5% was imported, whereas in 1939 the total consumption was 7,138,940 tons, of which 57.7% was imported. From the figures given it appears that the increased fuel consumption noted between 1914 and 1939 has been to a great extent met by the increase of petroleum produced within the country.

H. I. L.

1229.* **Adequacy of the Supply of Petrol for the whole of the Republic.** Y.P.F. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 14.—The Y.P.F. publish a notice giving an assurance that in spite of present difficulties in obtaining regular supplies of foreign products they are prepared to provide all the petrol that may be required in the country from the Government and private refineries. The public need therefore have no anxiety regarding the supply of petrol and, moreover, should a private company run short of petrol temporarily in any particular part, the Y.P.F. and its branches will meet their needs without delay.

H. I. L.

1230.* **Summary of the Work Carried Out in Refining Plants in the Argentine Republic during the year 1938.** Anon. *Bol. Inform. Petroleras*, Feb. 1940, XVII (186), 27-29.—This summary includes three tables. One contains a census of works, number of employees, number of workmen, compared with the two previous years and similar comparative data showing expenditure on labour, material and power supply. Table II deals in detail with the materials used for treatment of the petroleum in the refineries, and Table III provides particulars of the various products, both as regards quantity and sale values.

H. I. L.

BOOK REVIEW.

Geology of Eastern Iran. By F. G. Clapp. (*Bulletin of the Geological Society of America*, Vol. 51, 1940.) Pp. 101; 13 Pls.; 8 figs. Published by the Society, 50 Church Street, New York, N.Y.

The liquidation of the Amiranian Oil Co. has enabled its geologists to publish the general results of their surveys much earlier than had at one time been expected. Dr. Clapp's paper—the first, it is hoped, of a series of Amiranian origin—is a pioneer attempt to put down the high lights of a large tract of country the geology of which has been little more than an intriguing mystery for many decades. Those familiar with the delights of Iranian geology will not be deceived by the simple manner in which he presents his facts. They can appreciate the tremendous volume of travelling and field-work involved, often under conditions with which our more domesticated geologists are unfamiliar, and they will sympathize with the author in his formidable task of compiling and correlating such a mass of data. His own modest remark that his paper forms a "necessary background" for anything which may subsequently be written about Eastern Iran will be cordially endorsed by all who know the country.

With felicitous and characteristic astuteness, Dr. Clapp has disarmed possible critics in advance by confining himself rigidly to facts of observation and abstaining from all forms of deduction and theorizing. This is perhaps a pity, for his is an experienced and rational mind, and a few reflections on the geological history of his area, from a man of his calibre, would have garnished the almost Spartan fare of his facts.

On the structural side, he is content to present his observations and to leave the reader to reach his own conclusions, stimulated only by a brief reference to the views of the various authorities of earlier vintage—Suess (E.), Argand, Suess (F. E.), and de Böckh, Loes, and Richardson. On one point, however, it may be fairly observed here that his "true nappes of the Alpino type" (page 17 and Fig. 3, page 18) would scarcely be accepted by the Alpine school. It may be that these published profiles do not go deep enough to bring this feature out, but, on the other hand, it can safely be said that they bear little resemblance to the genuine decken of the Zagros country along the southern edge of the Iranian Plateau. A revision of Riviero's cross-sections of the Central Elburz may be forthcoming when the results of the survey work now being carried out by the Imperial Government are published. As seen on the ground, the profiles certainly do not suggest nappes.

A great wealth of stratigraphical knowledge is presented from the Elburz to Zahedan and Afghanistan, and no later revision of detail will impair the great value of this compilation for future investigators. Here again, however, it is felt that Dr. Clapp has perhaps been rather cautious and that a correlative summary of each formation—manifestly possible in some cases, at least—would have brought his great mass of stratigraphical information into better focus for his readers.

Pre-Devonian rocks appear to be as poorly developed in Eastern Iran as they are elsewhere in that country, but the Upper Palæozoic is well authenticated, even if the actual boundaries everywhere from Devonian to Trias are not always clear. Again, as elsewhere, the Trias is generally difficult to identify, unless in a calcareous facies, but the Jurassic attains considerable thicknesses on occasion, and its economic importance as the source of Iran's coal supplies (Lias) might have been given greater prominence by someone less interested in the rival form of fuel. The Cretaceous, at all times one of the most restless formations in the country, is well treated here, and perhaps it need only be added that the suspected volcanic phase between Lias and Cretaceous (page 54) can be clearly seen in the Central Elburz near Lahr, where, in several places, a variable thickness of green and red volcanic ash and tuff overlies now Lias, now Jura Limestone, and is in turn covered by dark

massive limestone with abundant Aptian Orbitolinas. The history of the vast and confused Tertiary basins is obscure. Work recently carried out in the Central Elburz by the Imperial Government shows that the "Green Beds" penetrate deeper into the column than had hitherto been believed—for example, in the high country north of Lahr the succession in ascending order is: (i) Orbitolina limestone, (ii) (andesitic) lavas, (iii) a mixed series of pyroclastics and (Middle) Eocene limestones, (iv) a considerable thickness of "Green Beds," (v) a second Nummulitic series the assemblage of which has not yet been worked out. The geological study of these Tertiary basins would be a fascinating exercise, but it lacks a commercial incentive at the present time.

It was perhaps too much to hope that Dr. Clapp would let fall from the rich table of his journeyings in Amirania some crumbs of interest to his fellow oil-men. He has, however, done the next best thing, and purveyed a sound and comprehensive factual report; and, for that, they are heavily in his debt. G. M. SHAW.

BOOKS RECEIVED.

The Mexican Oil Seizure. D. R. Richberg. Pp. 56. Standard Oil Co. (N.J.), 30 Rockefeller Plaza, New York City.

Mr. Richberg acted as counsel for the Oil Companies involved in the Mexican expropriation of foreign-owned oil properties, and in this pamphlet he endeavours to portray accurately the events leading up to the expropriation as well as the subsequent negotiations. To aid the reader in a clearer understanding of the position, the author devotes some pages to the historical background and describes the progress of the revolution in Mexico since 1913.

The writer offers no solution to the problem as it exists at present, apart from those already sought in vain, but, in his own words, has written this narrative "to aid in public understanding of the causes of a continuing controversy of profound interest" so that . . . "in the long run public opinion adequately informed should arrive at a sound judgement of what ought to be done."

Present Status of the Mexican Oil "Expropriations" 1940. Pp. 79. Standard Oil Co. (N.J.), 30 Rockefeller Plaza, New York.

The attempt of Mexico to confiscate the subsoil petroleum is not new, various devices having been tried to accomplish this since 1917. The United States has, however, uniformly resisted the effort, and the record of that resistance is set out in this memorandum. During the past year the Oil Companies have endeavoured to negotiate a private settlement with the Mexican Government, and a brief record of the negotiations is also given. The memorandum endeavours to show that the unsuccessful termination of the private negotiations now requires the interposition of the United States Government to secure Mexico's compliance with her obligations under international law.

Roads and Road Construction Year Book and Directory, 1940-41. 10s. 6d. Carriers Publishing Company, 68 & 69 Shoe Lane, London, E.C.4.

This, the ninth, edition of *Roads and Road Construction Year Book and Directory* contains more than 400 pages of information useful both to the road engineer and to the technologist who is interested in materials for road construction.

Nearly a third of the book is devoted to a review of Road Development in 1939 and a Survey of Current Practice, including a Bibliography of recent literature on road-making, etc. These sections are followed by comprehensive lists of names of Highway Officials, Manufacturers and Suppliers of Road-making Materials, Road Contractors, and Proprietary Names.

ASLIB War-Time Guides to British Sources of Specialized Information. No. 1. Fuel and Allied Interests (excluding Electricity). June 1940. Association of Special Libraries and Information Bureaux, 31 Museum Street, London, W.C.1. Price 2s. to Members of ASLIB, 2s. 6d. to Non-Members.

This useful guide to British sources of specialized information relating to the Coal, Petroleum, Gas, and Gas-coke Industries is arranged in seven sections. The first section is devoted to a description of the war-time organization of these industries; Section 2 gives a list of sources of specialist information relevant to Fuel and allied interests; Section 3 lists periodicals publishing abstracts or lists of references; Section 4 covers trade and technical periodicals; Section 5 comprises publications issued by professional organizations and industrial concerns; Section 6 lists annual publications. Subject indexes to sources of information and to publications are given in the final section.

British Standard No. 903—1940 Methods of Testing Vulcanized Rubber. Pp. 74. British Standards Institution, 28, Victoria Street, London, S.W.1. Price 5s. net.

The British Standard Methods of Testing Vulcanized Rubber—*i.e.*, the product obtained from natural raw rubber by heating it with sulphur and/or some sulphur-containing compounds—have been based on those of the Research Association of British Rubber Manufacturers.

Full details are given of methods of chemical analysis of vulcanized rubber and of methods of physical testing of soft vulcanized rubber and ebonite. The methods of test are precision standards of reference, and it is pointed out that it is not expected that all the refinements of detail will be applied in day-to-day routine test work.

Directory of British Fine Chemicals. Produced by Members of the Association of British Chemical Manufacturers, 166 Piccadilly, W.1. Pp. 79. April, 1940. (*Distributed gratis to bona fide users of fine chemicals on direct application to the above address.*)

This publication, comprising a list of fine chemicals manufactured in Great Britain together with an index to manufacturers, supersedes the edition published in April 1935.

The Motor Transport Yearbook and Directory. 24th Annual Volume. 1939-40. Pp. 786. Electrical Press, Ltd., 60, Kingsway, London, W.C.2. Price 30s.

The classification of the contents of this volume follows that of previous editions, and consists of a Statistical Survey and four main sections. "Road Transport Organization" is the title of a section dealing with law and other data relating to licensing, taxation, construction, equipment, maintenance, and use on roads of motor vehicles for public passenger and goods transport. Section I is devoted to a comprehensive survey—financial, commercial and descriptive—of motor transport undertakings. Section II provides financial and commercial information of the manufacturing companies which produce the vehicles and accessories required for the transport services. Section III comprises the names and addresses of the directors and officials engaged in the undertakings described in the *Year Book*.

Statutory Rules and Orders, 1940. No. 749. Emergency Powers (Defence). Petroleum Department. Pp. 2. H.M.S.O., York House, Kingsway, London, W.C.2. Price 1d.

Relates to the establishment of a Petroleum Department of the Board of Trade under a Secretary for Petroleum.

Statutory Rules and Orders, 1940. No. 826. Emergency Powers (Defence). Motor Fuel Rationing. Pp. 5. H.M.S.O., York House, Kingsway, London, W.C.2. Price 2d.

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ANNUAL REVIEWS OF PETROLEUM TECHNOLOGY

Volume 5.

Volume 5 of the series of *Annual Reviews of Petroleum Technology* is published at the beginning of September 1940. It covers developments in the industry during 1939 and the early part of 1940. The published price is 11s., and members of the Institute and subscribers to the *Journal* may purchase one copy at 5s. 6d. (post free to United Kingdom and overseas). A prospectus of the book is included with the present number of the *Journal*.

A reduced number of copies of the book has been printed owing to restrictions on the consumption of paper, and members wishing to obtain a copy are asked to order as soon as possible. (This does not apply to members who have a standing order for all volumes in this annual series.) In order to save book-keeping and postage, members are asked to send a remittance with their order. Members in North and South America may remit by drafts in dollar currency.

The *Annual Reviews* constitute the only complete summary available of the whole field of literature relating to petroleum technology, and the Council hopes that members will give their full support to this important publication.

CANDIDATES FOR ADMISSION.

The following have applied for admission to the Institute or transfer to another grade of membership, and in accordance with the By-laws the proposals will not be considered until the lapse of at least one month subsequent to the issue of this *Journal*, during which any Fellow, Member, or Associate Member may communicate by letter to the Secretary, for the confidential information of the Council, any particulars he may possess respecting the qualifications or suitability of any candidate.

The object of this information is to assist the Council in grading candidates according to the class of membership.

The names of the candidate's proposer and seconder are given in parentheses.

BARRS, Gerrit, Refinery Engineer, Bataafsche Petroleum Maatschappij. (J. A. Oriel; E. Le Q. Herbert.)

DAVIDSON, Anthony Dryden, Chemical Engineer, Foster Wheeler, Ltd., London. (A. W. Nash; R. K. Fischer.)

HOLE, Herbert Wray, General Manager, New Brunswick Gas and Oilfields, Ltd., Moncton, New Brunswick. (C. A. P. Southwell; A. E. Dunstan.) (Trans. from A.M.)

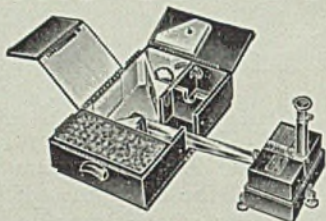
JOHNSTON, Douglas Gordon, Engineer, Texas Oil Co., London. (N. L. Anflogoff; W. A. Webster.)

KEACH, Raymond Harry, B.Sc., Chemical Engineer, Foster Wheeler, Ltd., London. (A. W. Nash; R. K. Fischer.) (Trans. from Stud.)

ARTHUR W. EASTLAKE,

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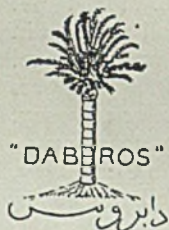


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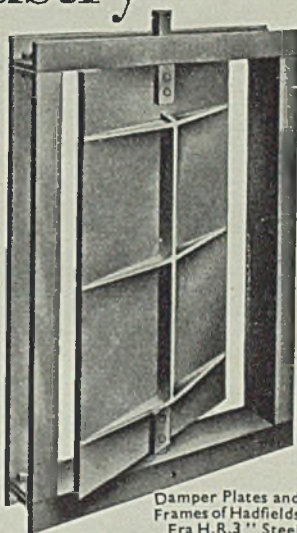


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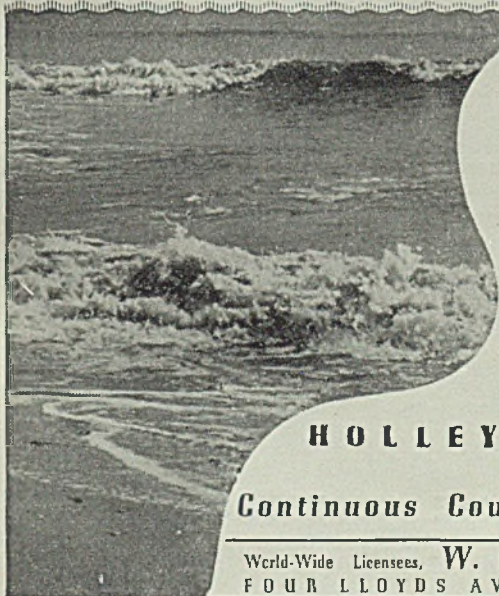
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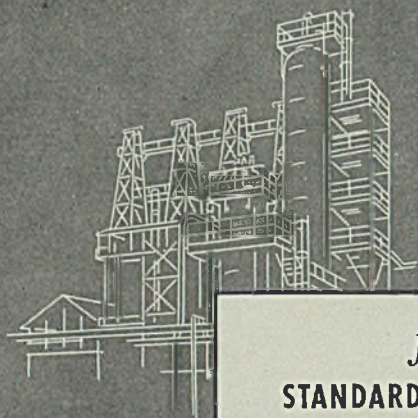
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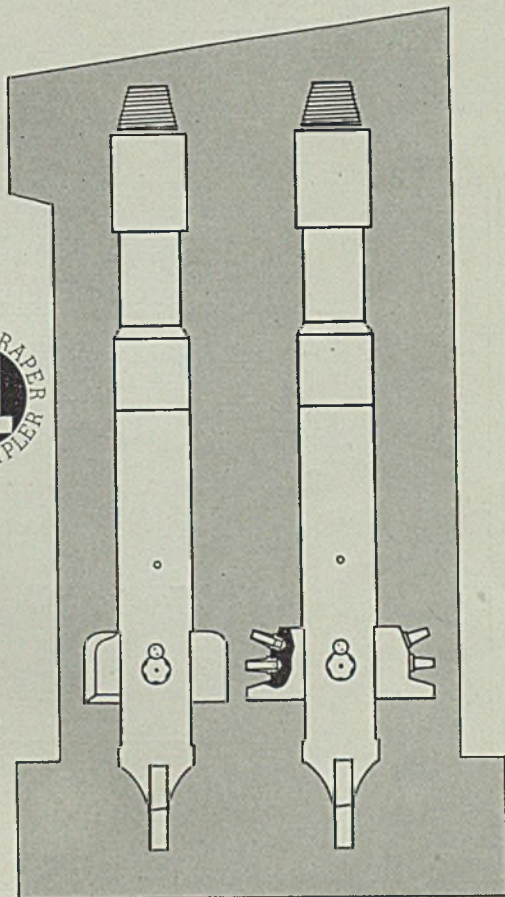
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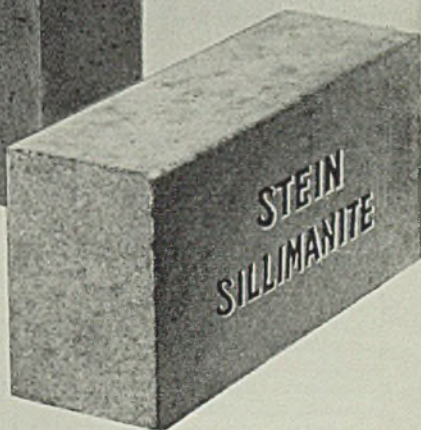
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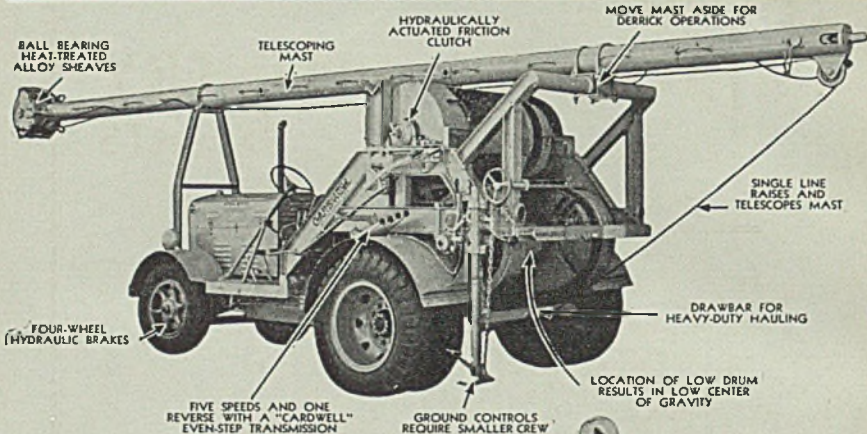


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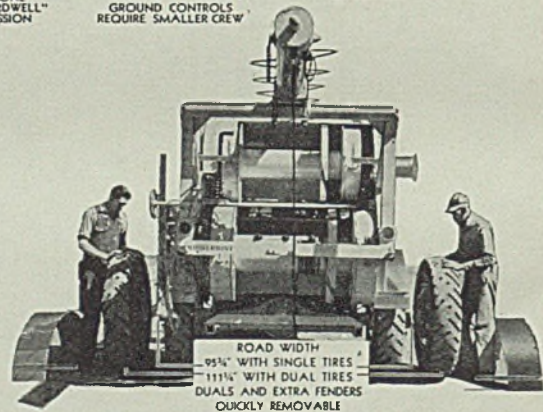
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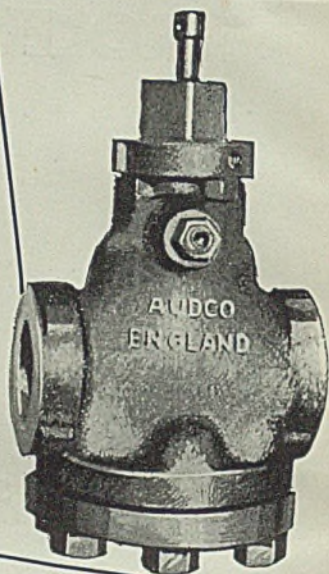
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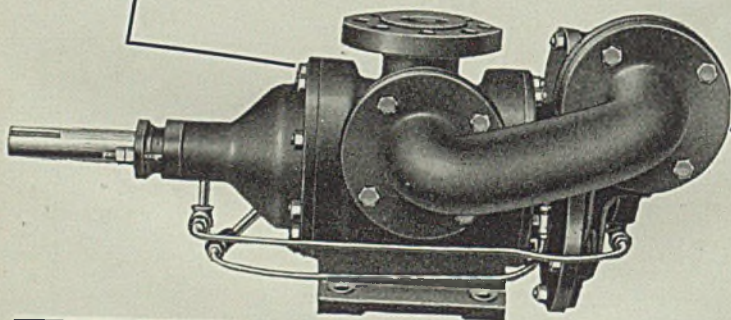
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