VOL. 27. NO. 207. JANUARY 1941.

STUDIES ON THE SEPARATION OF PARAFFIN WAXES. PART III.—THE SWEATING OF OIL-FREE WAXES.*

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

THE production of paraffin wax from wax distillate may be considered to take place in the following stages :—

(1) Partial removal of the oil by chilling and filter pressing ;

(2) Removal of oil and waxes of low melting point from the wax cake by the process of fractional melting—the so-called sweating process ; and

(3) Physical or chemical refining of the sweated wax.

It has previously been stated[1] that this research project is concerned primarily with the sweating stage, the principle of this operation being based on the fact that the various waxes comprising the slack wax have progressively higher melting points. The fractions removed contain valuable products, and re-sweating is therefore an important feature of the commercial process. Innumerable combinations of re-sweating are possible to give a wide range of products differing in melting point and oil content, but the investigation of this is outside the scope of the present paper, which is concerned with the fractional melting of oil-free waxes.

The efficiency of the sweating or fractional melting operation is dependent on some or all of the following factors :—

(1) The sweating equipment.

(2) The rate of sweating.

(3) The size and shape of the wax crystals, which may be affected by—

(*a*) Composition of the wax.

(*b*) Amount of oil present in the oil–wax mixture comprising the slack wax.

(*c*) Viscosity of the oil ; and

(*d*) Treatment of the wax–oil mixture prior to sweating (*e.g.*, the crystal size may be affected by the rate of cooling in the sweater).

It should be noted, however, that there is no general agreement that all the above factors contribute towards the efficiency of the process. It is clear that if a single wax stock and the same sweater are employed throughout the investigation, the effects of the following variables must be considered :—

(1) Rate of sweating ;

(2) Rate and extent of cooling of the wax or wax–oil mixture in the sweater prior to commencing the sweating operation ;

---

* Paper received 21st November, 1940.

(3) Amount of oil in the wax–oil mixture;
(4) Viscosity of the oil.

The process may be operated in different ways: a uniform temperature increase may be employed throughout the sweating operation; a period of gradual temperature rise may be combined with periods of constant temperature at different stages; the rate of temperature increase may be so regulated that the amount of liquid product collected in a given time remains constant for the duration of the experiment.

A consideration of the above methods of operation in conjunction with the design of the laboratory sweating apparatus indicated that the last method was the most likely to lead to accurate control, and was therefore likely to give the most satisfactory comparative results.

SWEATING EQUIPMENT.

Satisfactory sweating, besides being dependent on the nature of the wax, is especially sensitive to the adequate control and regulation of temperature throughout the system, and to an adequate means of draining the liquid formed during the operation. It was believed that an electrically heated sweater of the stove type would be most likely to fulfil these requirements in laboratory scale-experiments.

The sweater finally employed consisted essentially of a cage in which the wax was supported, fitted with a series of internal heating coils and also a surrounding external heater. The component parts are shown in Fig. 1. The cage *A* is a cylinder of length 6½ inches, diameter 3¼ inches, open at one end, and made from perforated brass sheeting having forty-two perforations (of diameter $\frac{7}{64}$ inch) to the square inch. The spacer or carrier *B*, which fits into the open end of the cage, consists of two circular brass discs 1½ inches apart. Each disc contains three small holes for the insertion of thermometers, and nineteen holes each of diameter ⅜ inch arranged symmetrically over the disc. The chief function of the spacer is to maintain the nineteen internal heaters *C* evenly spaced in the cage, this even spacing of the heaters being essential in order to secure a uniform distribution of the heat throughout the wax mass in the sweater. Each of the internal heaters consists of an outer glass tube of length 7½ inches and diameter $\frac{5}{16}$ inch, sealed at one end, which contains the heating element comprising 2 feet 6 inches of resistance wire (resistance 1·08 ohms per foot), supported on a length of glass tube of considerably smaller diameter.

A thickness of asbestos paper was placed between the heater and the external glass tube, and a small amount of asbestos powder was placed in the bottom of this tube. The heating elements were joined in series with small terminal connections.

The internal heating tubes, supported in the spacer, made contact with the bottom of the cage when placed in position in the latter. The set-up of the sweater when ready for use is shown in Fig. 2. The external heater (not shown) consisted of an iron sheet welded into the form of a hollow cylinder of length 10½ inches and diameter 4¼ inches. The outer surface was covered with a layer of asbestos on which was wound the heating element, which, in turn, was covered with a thicker layer of asbestos.

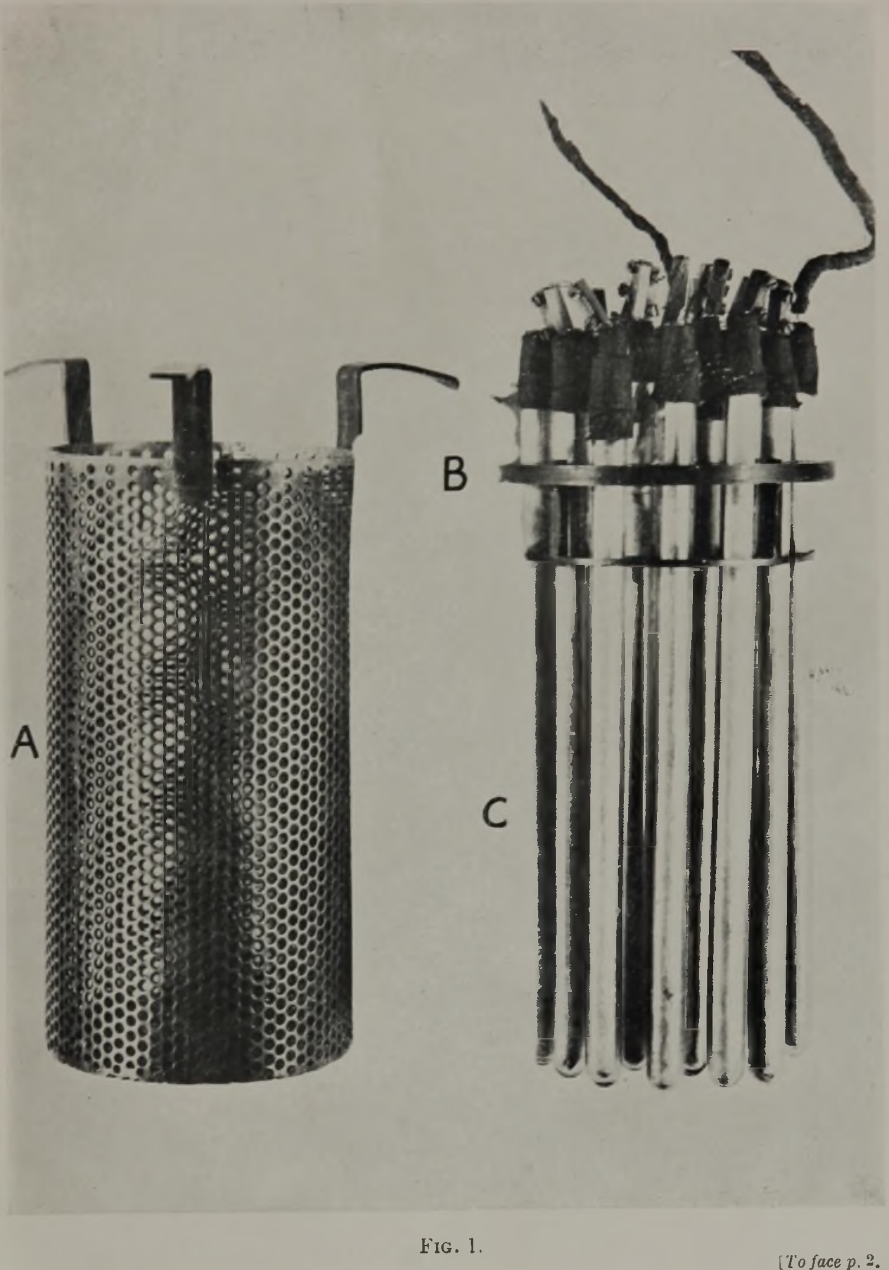


FIG. 1.

[To face p. 2.

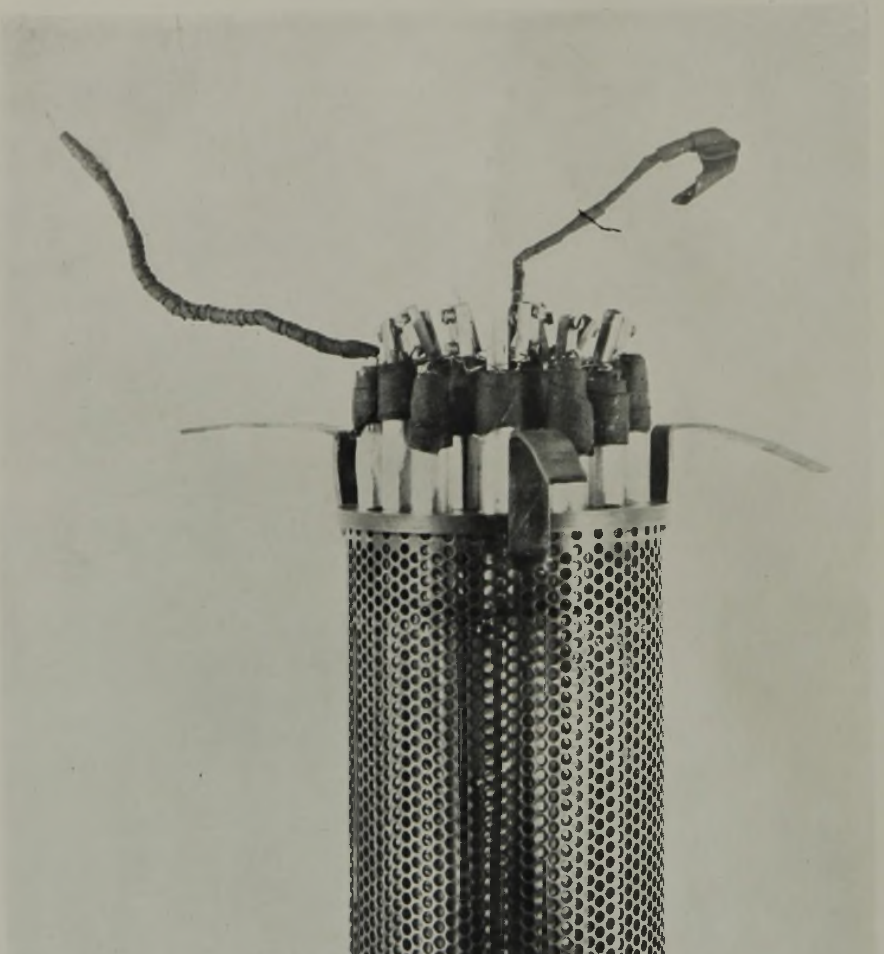


FIG. 2.

After this work had been in progress for some time it was found that a laboratory sweater of similar construction had been previously developed by the Research Staff of the Burmah Oil Company.

EXPERIMENTAL PROCEDURE.

The following method of preparing the sample was found to be very satisfactory. The outer surface of the sweater was completely covered with a sheet of cellophane held tightly against the surface by means of a number of rubber bands. Additional support was afforded the cellophane covering the bottom of the sweater by placing the latter in a suitable tin-lid containing a small amount of molten wax; the wax was solidified by placing the bottom of the apparatus in cold water. In order to heat the glass containing the internal heating elements, current was passed through the latter for a short period, after which the molten wax sample, which had been maintained at a temperature 15–20° F. above its melting point, was introduced into the apparatus through a glass filter funnel inserted into one of the thermometer holes in the spacer. The cage was filled to within ½ inch below the lower plate of the spacer, the capacity being approximately 440 grams of wax. The sweater and its charge were then placed in a position away from draughts and allowed to cool under any desired conditions. Cooling of the mass to room temperature normally occupied about 6 hours. At the end of this period the lower portion of the apparatus was momentarily placed in warm water to loosen the wax adhering to the tin; the cellophane covering was then taken from the surface of the sweater and the very thin layer of wax partly covering the latter was removed.

For the actual sweating or fractional melting experiment the external heater was supported on a tripod in which was placed a glass filter funnel of diameter 4 inches (*i.e.*, of larger diameter than the sweater). A screen of asbestos paper was placed round the tripod. The sweater was supported in the centre of the external heater. During the experiment the liquid wax phase fell from the sweater into the filter funnel, and thence into any convenient collecting vessel. It was found, especially during the later stages of the experiment when the liquid phase consisted of waxes of comparatively high melting point, that the liquid tended to solidify in the filter funnel. This was prevented by maintaining a minute gas flame in a position between the beaker and one of the tripod legs—the warm air from the flame being deflected on to the filter funnel by means of a small sheet of tin held in a clamp.

The simple electrical heating arrangement employed is shown in Fig. 3, and is self-explanatory. There are two separate circuits :—

(1) *Internal heating*, comprising a variable resistance R_1, fixed resistances R_3 and R_4; ammeter A_1 (range 0–250 milliamp.); and the internal heating elements of the sweater ; and

(2) *External heating*, comprising a variable resistance R_2, fixed resistances R_5 and R_6; ammeter A_2 (range 0–1 amp.); and the external heater.

Little difficulty was encountered in maintaining any desired rate of sweating between 3 per cent. and 15 per cent. per hour, but at rates higher than 15 per cent. per hour the process tended to become unmanageable. With care and constant attention it was possible to limit the temperature gradient through the wax mass to 2–3° F. for oil-free waxes, and to 1° F. for waxes containing oil.

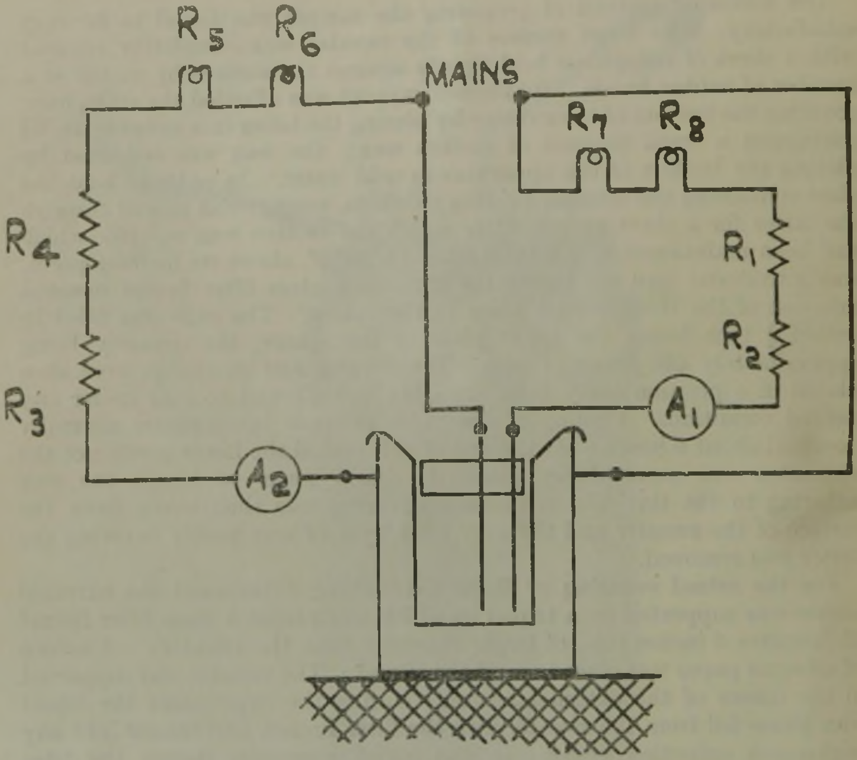


FIG. 3.

The external heating was regulated so that the air temperature in the space between the sweater and the external heater was within ± 0·5° F. of the temperature of the liquid dropping from the sweater.

EXPERIMENTAL STUDY OF THE FRACTIONAL MELTING OF OIL-FREE WAX.

Employing a given oil-free wax stock, two only of the variables previously mentioned have to be considered: the rate of sweating, and rate and extent of cooling of the wax prior to sweating. The time–cost factor is of obvious industrial importance, but this variable cannot receive consideration here.

The object of the sweating process is to remove oil, and to obtain as high a yield as possible of a satisfactory wax of some desired melting point, and hence, in the absence of oil, the latter becomes the principal object.

For the purpose of investigating the effect of the rate of sweating on the yields of products, a series of experiments covering the range 3–20 per cent. per hour was made.

The wax stock, melting point 122·1° F., was that used in the determination of the wax equilibrium diagram,[2] and the sample used in each experiment was prepared according to the procedure already described, and then allowed to solidify over-night. The temperature of the wax mass was gradually increased from room temperature, over a period of 4–4½ hours, before the initial appearance of the liquid in the collecting vessel. From this stage the heating was arranged so that the fractional melting proceeded at the desired rate.

The first and, on occasion, the second fractions—representing a 5–10 per cent. yield were collected at a slightly slower rate, this precaution being taken to avoid the possibility of the experiment getting out of control in the early stages. By the time 10 per cent. of the stock had been collected, the fractional melting was proceeding at the desired rate.

When less than 5 per cent. of the stock remained in the sweater, the current passing through the internal and external heaters was increased, in order to facilitate the removal of this residue wax. The last traces were removed by supporting the sweater in an air oven maintained at a temperature of 150–160° F., and allowing the last drops to fall into the collecting vessel. The draining was assisted by occasionally shaking the apparatus, and, by following this procedure, the amount of wax finally remaining in the sweater was seldom more than 1 gram.

When the liquid phase was removed at rates of 10 per cent. per hour and higher, the experiment was completed in 1 day, but for rates of 5 per cent. and 3 per cent. per hour, 2 and 4 days, respectively, were required for the completion of the runs. During the first day of the 5 per cent. per hour runs, 50–60 per cent. of the wax was removed from the sweater, and that remaining in the apparatus was allowed to cool to room temperature over-night. On the second day the wax mass was slowly warmed over a period of 4½–5 hours before the initial drops of liquid phase were collected.

By careful regulation of the heating it was possible to control the rate to ±0·5 per cent. per hour except in the 20 per cent. per hour run, where the rate was too high for accurate control.

A temperature gradient of 2–3° F. normally existed in the wax mass during the first half of the run when larger quantities of wax were present in the sweater. As the experiment progressed, however, this temperature gradient became smaller.

Difficulty was experienced in maintaining the desired rate during the fractionation of the last 10 per cent., owing to the comparatively small amount of wax present in the sweater at this stage. Any errors involved are, however, believed to be small and relatively unimportant.

The sweats were, in general, collected in cuts of 5–7 per cent. by weight, and the melting point of each fraction was determined in the manner previously described.[2]

In order to obtain the figures for a comparison of the relative efficiencies for the different rates of separation of the waxes, it is necessary to know the yield and melting point of the residual wax present in the sweater at any given stage of the experiment. This may be found by :—

(*a*) Blending the fractions—starting with the last two collected—and determining the melting point of the blend after the addition of each two consecutive fractions ; or

(*b*) Calculation, employing the relationship $T = \frac{414{\cdot}5\,M}{M + 94{\cdot}4}$ as previously described.

Excellent agreement was found between experimental and calculated figures, as indicated in columns 8 and 9 of Table I, which gives the essential data obtained for the 3 per cent. per hour run.

Table I.

Experimental Run No. 8. *Rate of Sweating,* 3 *per cent. per hour.*

Fraction No.	Liquid phase.				Residue wax in sweater.			
	Wt., gms.	Yield, wt. % of stock.	M. pt., ° F.	Mol. wt.*	Yield, wt. %.	Mol. wt.*	M. pt. (calc.), ° F.	M. pt., ° F.
0	—	—	—	—	100·0	333·7	122·00	122·05
1	18·68	4·8	106·25	296·2	95·2	335·6	122·65	—
2	21·92	5·6	109·05	302·3	89·6	337·6	123·45	123·50
3	20·52	5·3	110·00	304·6	84·3	339·8	124·35	—
4	26·78	6·9	112·60	310·5	77·4	342·3	125·20	125·20
5	22·58	5·8	113·75	313·2	71·6	344·6	126·05	—
6	24·91	6·4	116·50	319·9	65·2	347·1	127·05	127·00
7	23·80	6·1	118·35	324·5	59·0	349·4	127·90	—
8	22·94	5·9	119·40	327·0	53·1	351·9	128·85	128·90
9	23·02	5·9	120·20	329·2	47·2	354·8	129·90	—
10	23·16	6·0	121·10	331·5	41·3	358·2	131·10	131·20
11	23·11	5·9	123·35	337·3	35·3	361·7	132·30	—
12	26·28	6·8	125·75	343·7	28·6	365·9	133·80	133·80
13	26·60	6·8	127·05	347·1	21·7	371·8	135·75	—
14	22·92	5·9	130·45	356·5	15·8	377·7	137·70	137·75
15	27·47	7·1	133·65	365·5	8·8	387·5	140·65	—
16	13·08	3·4	137·30	376·5	5·4	394·2	142·70	142·70
17	20·62	5·4	142·70	394·2	0·0	—	—	—

* Calculated from the relation $T' = \frac{414{\cdot}5\,M}{94{\cdot}4 + M}$.

The respective yield/melting-point graphs for the 3 per cent. per hour run and the 14-stage equilibrium experiments [2] are shown in Fig. 4, and although the curves are of a similar form, it will be noted that for a given melting point the yield of wax is higher in the actual sweating process than in the multi-stage equilibrium experiments. This finding does not, however, affect in any way the theory developed in connection with the equilibrium diagram, but rather emphasizes the experimental difficulties involved in its determination. It is clear that, at least in respect to yields, the actual sweating experiment represents a nearer approach to the ideal or infinite-stage process than does the equilibrium experiment.

In the multi-stage equilibrium experiments it was found that the melting point of the liquid phase was, in general, 0·2–0·3° F. lower than the equilibrium temperature. It is therefore of interest to compare the melting points of the individual fractions obtained in the 3 per cent. per hour run with the temperature at which they left the sweater. The relevant figures are given in Table II.

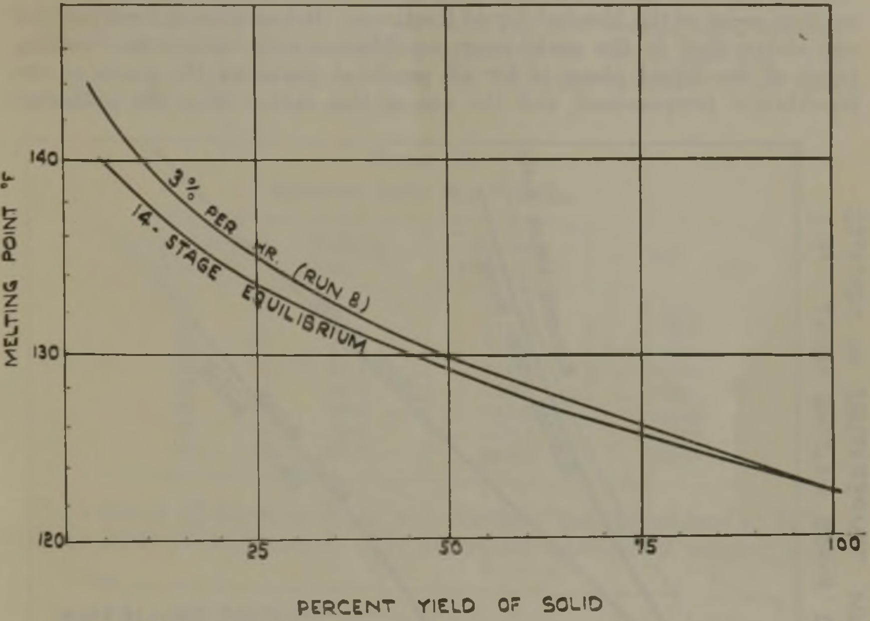


Fig. 4.

Table II.

Fraction No.	Melting point, ° F.	Temperature of wax at end of cut, ° F.	Difference in temperature, ° F.
1	106·75	112·7	5·9
2	109·05	114·8	5·7
3	110·00	117·3	6·3
4	112·60	118·0	5·4
5	113·75	118·6	4·8
6	116·50	120·0	3·5
7	118·35	121·9	3·5
8	119·40	124·2	4·8
9	120·20	124·8	4·6
10	121·10	126·0	4·9
11	123·35	128·0	4·6
12	125·75	129·6	3·8
13	127·05	132·4	5·3
14	130·45	135·0	4·5
15	133·65	139·0	5·4
16	137·30	144·0	6·7
17	142·70	—	—

The difference is considerable, and is probably due to drainage lag.

In Fig. 5 the equilibrium diagram for the 14-stage process is compared with the diagram resulting when the data obtained in the 3 per cent. per hour run are plotted in the same way. The equilibrium temperature, taken as the melting point of the liquid fraction, is plotted against the melting point of the solid remaining in the sweater and also against the melting point of the blended liquid fractions. It has already been pointed out above that in the multi-stage equilibrium experiments the melting point of the liquid phase is for all practical purposes the same as the equilibrium temperature, and the use of this rather than the indicated temperature at which the liquid fraction left the sweater is to be preferred. As will be seen from this figure, the two diagrams are closely similar. The 3 per cent. per hour run represents a nearer approach to the ideal infinite-stage process than does the multi-stage equilibrium experiment.

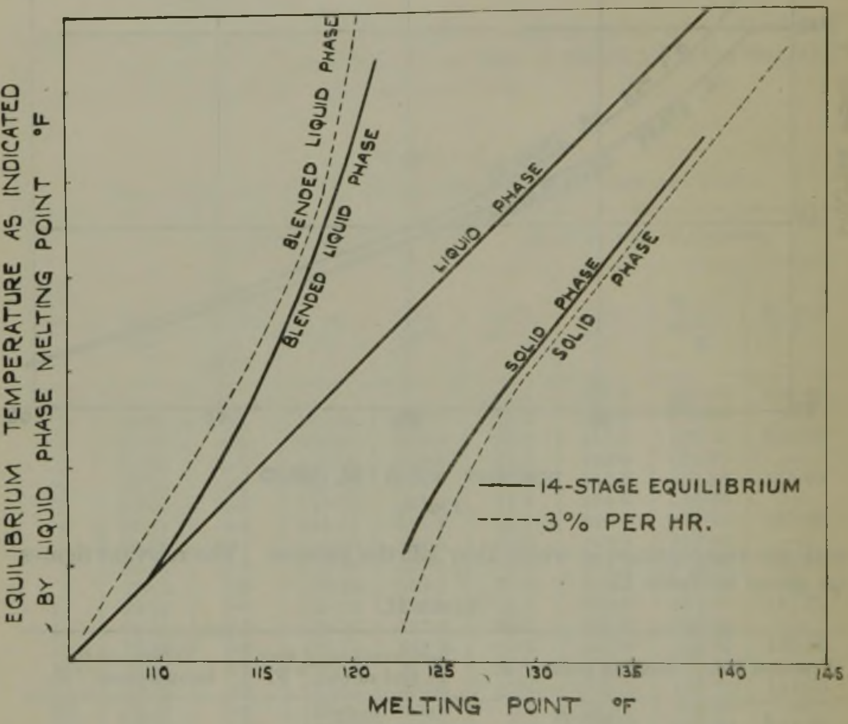


Fig. 5.

It is probable that the difference between the two diagrams is due to the difference between a 14-stage and an infinite-stage process. If the 14-stage equilibrium experiment could have been replaced by 30- or 40-stage equilibrium experiments, then the resulting diagrams would probably have been even nearer the ideal process diagram. It is clear from Fig. 5 that the previously derived conception of the sweating process is a true one, and that an equilibrium diagram based on the results of static experiment with a large number of stages could be used as a basis for computing sweating efficiency. In view, however, of the difficulty of carrying out such an experiment, it is suggested that the diagram obtained at a low rate of sweating—say, 3 per cent. per hour or less—in an experimental sweater such as that described in this paper, be used for this purpose.

The melting point/refractive index (n_D^{60}) relationship for the single fractions collected in the 3 per cent. per hour run is of special interest when compared with the corresponding relationship for the *n*-paraffins. The lower curve in Fig. 6 is plotted from the following values of n_D^{60} for pure *n*-paraffins taken from various sources.

TABLE III.

Refractive Index of n-*Paraffins.*

Carbon atoms in molecule.	Melting point, ° C.	Melting point, ° F.	n_D^{60}.
21	40·5	104·9	1·4287
22	44·4	111·9	1·4305
23	47·7	117·9	1·4319
24	51·1	124·0	1·4323
25	53·8	128·8	1·4341
26	56·6	133·9	1·4355
27	59·5	139·1	1·4366
28	62·0	143·6	1·4375

The value of n_D^{60} for each of the wax fractions was determined at 60° C. with an Abbé Refractometer. The values for fractions of melting point higher than 60° C. were determined at a temperature 1° C. above the respective melting points, and a correction factor of 0·0004 per 1° C. applied to obtain the equivalent value of n_D^{60}. The figures for n_D^{60} given in Table IV, from which the upper curve of Fig. 6 is plotted, are correct to ± 0·0001.

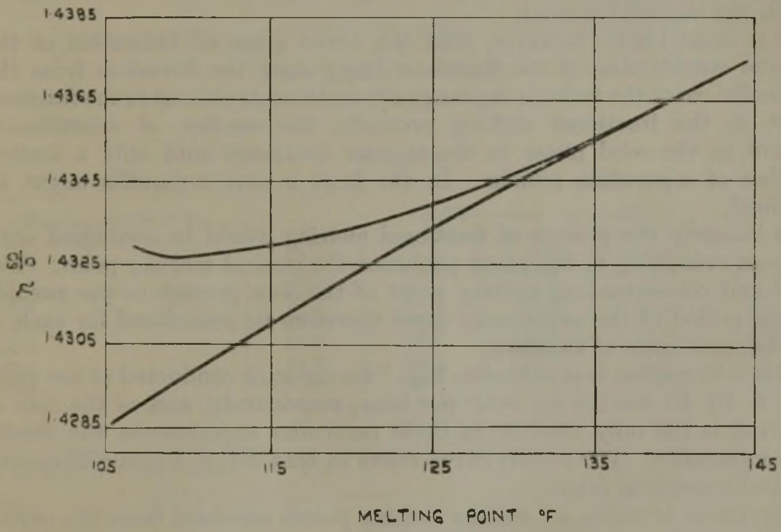


FIG. 6.

TABLE IV.

Fraction No.	Melting point, ° F.	n_D^{60}.	Fraction no.	Melting point, ° F.	n_D^{60}.
1	106·25	1·4331	9	120·20	1·4336
2	109·05	1·4327	10	121·10	1·4337
3	110·00	1·4328	11	123·35	1·4339
4	112·60	1·4328/9	12	125·75	1·4342
5	113·75	1·4330	13	127·05	1·4344
6	116·50	1·4332	14	130·45	1·4349
7	118·35	1·4333	15	133·65	1·4354
8	119·40	1·4334/5	16	137·30	1·4362
			17	142·70	1·4373/4

The curve for the wax is initially displaced from the corresponding *n*-paraffin curve, but approaches the latter as the melting point of the fraction increases. The two curves meet at the point representing Fraction No. 15, and then become almost coincident. The value of n_D^{60} for the first fraction is higher than for the second, an anomaly that may be explained by the presence in the first fraction of the small amount of oil originally present in the wax stock.

Two possible explanations may be advanced to account for the nature of the curve for the wax fractions.

The values of n_D^{60} for *iso*paraffins are considerably higher than for *n*-paraffins of the same melting point. It is therefore possible that the wax contains *iso*paraffins which are gradually removed during the process of fractional melting until removal is complete at Fraction 15, the stage at which the two curves meet.

It is more likely, however, that the curve gives an indication of the relative complexities of the fractions, the greater the deviation from the *n*-paraffin curve the wider is the range of constituents present in the fraction. Thus, as the fractional melting proceeds, the number of constituents present in the solid phase in the sweater decreases until only a limited number of *n*-paraffins remain. In the limit a pure *n*-paraffin might be obtained.

In industry the process of fractional melting would be continued until the wax remaining in the plant possessed the desired melting point. The yield and corresponding melting point of the wax present in the sweater at any period of the experiment must therefore be considered for each of the different rates of sweating.

This information is provided in Fig. 7 for the runs conducted at the rates of 3, 5, 10, 15 and 20 per cent. per hour, respectively, and as the rate of sweating is the only variable in these particular experiments, the results are comparable. The dotted curve refers to Run No. 4, which will receive particular mention later.

The yields of waxes of various melting points obtained from the curves in Fig. 7 are given in Table V for each of these runs.

For rates of 10 per cent. per hour and higher the yields of wax when less than 10 per cent. should be accepted with some reserve, as they are probably too high, owing to the difficulty experienced in maintaining the high rate of sweating with only a small amount of wax remaining in the sweater.

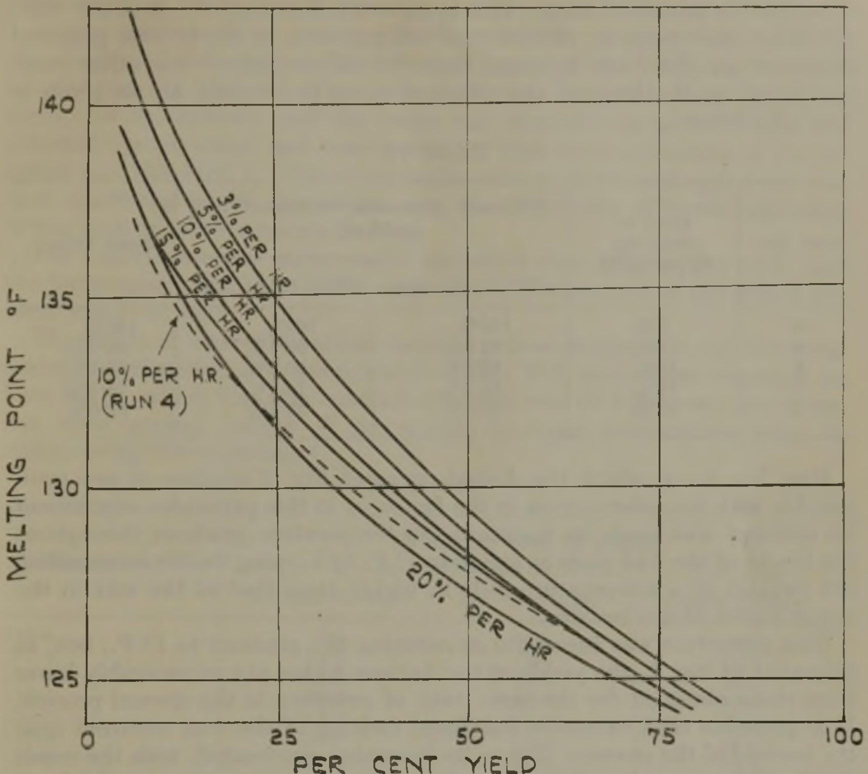


FIG. 7.

TABLE V.

Melting point, ° F.	Yield per cent. (by weight).				
140·0	10·4	7·4	3·0	—	3·1
137·5	16·5	13·5	8·8	7·0	7·0
135·0	24·2	21·2	17·2	14·8	13·1
132·5	34·4	31·0	27·5	25·7	20·5
130·0	47·0	42·9	40·5	38·3	30·8
127·5	61·5	58·0	54·8	55·2	47·5
125·0	78·8	75·8	74·3	75·0	69·8
Run No.	8	6	3	2	7
Sweating rate (per hour).	3%	5%	10%	15%	20%

It is clear from the data contained in Table V that the yield of wax of a given melting point decreases with increase in the rate of sweating. The yields of 130° F. and 132·5° F. melting-point waxes may be taken as a satisfactory basis for the estimation of the relative efficiencies at the different rates, as these waxes are present in the sweater in each experiment at a stage when the sweating has been proceeding at the desired rate for a considerable period of time. If it is assumed that Run No. 8 (3 per cent. per hour) represents an efficiency of 100 per cent. as the nearest practical approach to the ideal process, then the efficiencies of the other runs, calculated on the basis of the yields of these two waxes, are as given in Table VI below :—

TABLE VI.

Run No.	Rate of sweating, % per hour.	Efficiency of process calculated from yield of :		Mean value.
		130° m. pt. wax.	132·5° m. pt. wax.	
8	3%	100%	100%	100%
6	5%	91·3%	90·1%	90·7%
3	10%	86·2%	80·0%	83·1%
2	15%	81·5%	74·7%	78·1%
7	20%	65·5%	59·6%	62·5%

Run No. 4—to which the dotted curve of Fig. 7 applies—is not comparable with the other curves in the figure, as in this particular experiment an attempt was made to maintain the temperature gradient throughout the length of the wax mass at less than 1° F. by keeping the air surrounding the sweater at a temperature 2–3° F. higher than that of the wax in the upper region of the sweater.

This procedure was successful in reducing the gradient to 1° F., but, as indicated in Fig. 7, the yields of the various waxes are considerably lower than those obtained for the same rate of sweating in the normal process. It is probable that excessive fractional melting of the wax occurred near the surface of the sweater, due to its becoming overheated, with the result that uniform sweating of the wax throughout the mass was not obtained. Local overheating therefore has an adverse effect on the yields of the various waxes.

EFFECT OF METHOD OF PREPARATION OF SAMPLE PRIOR TO SWEATING.

Two experiments—runs 10 and 11—were conducted for the purpose of investigating the extent to which the efficiency of the process is dependent on the method of preparing the wax sample prior to sweating.

In run No. 10 the sweater was prepared in the normal manner and the wax was introduced at a temperature of 135–140° F. The sweater and contents were immediately plunged into an acetone–solid CO_2 mixture at $-55°$ C. The acetone was maintained at a temperature of -50 to $-55°$ C. during the solidification of the wax, which was complete in 35 minutes. After solidification, the wax temperature fell at the rate of 3° F. per minute, and when the central region of the mass had cooled to 50° F., the sweater was removed from the acetone bath. The sample remained at room temperature over-night, and sweating at the rate of 5 per cent. per hour was commenced on the following day.

During the early stages of the run some difficulty was experienced in keeping the temperature gradient of the wax mass within 3° F., but the run proceeded normally after the collection of the initial 10 per cent.

For run No. 11 the sweater was prepared in the normal manner and the wax was introduced at a temperature of 135° F. Cooling took place in the absence of draughts until the central region of the wax mass was at a temperature of 110° F. The cellophane was removed from the outer surface of the sweater, and the latter was immediately placed inside the external heater, which had been heated so that the temperature of the air inside was 105–110° F. Sweating at the rate of 5 per cent. per hour was then conducted in the normal manner, the first drops of liquid appearing in the receiver after 1 hour's heating.

The experiment was more easily controlled than in previous runs, and the temperature gradient in the mass after the collection of the first 5 per cent. was 1° F. only.

The yields of waxes of various melting points obtained in the two runs under consideration are compared in Table VII with those obtained in Run No. 6, which was also conducted at the rate of 5 per cent. per hour, but after normal cooling of the sample to room temperature prior to commencing the experiment.

TABLE VII.

Melting point, ° F.	Yield, per cent. by weight.		
	Normal cooling.	Partial cooling.	Shock cooling.
140·0 . . .	7·4	6·8	6·2
137·5 . . .	13·5	12·0	12·0
135·0 . . .	21·2	18·8	18·0
132·5 . . .	31·0	28·7	27·6
130·0 . . .	42·9	41·7	39·2
127·5 . . .	58·0	57·4	54·0
125·0 . . .	75·8	75·0	73·2
Run No. . .	6	11	10
Rate . . .	5% per hour	5% per hour	5% per hour

The yields of the various waxes obtained in Runs 6 and 11 are very similar, and it appears that only a slight advantage is to be gained by cooling to room temperature before commencing to sweat. It is likely that this slight increase in yield would be more than balanced in the industrial operation by the increased cost involved in the additional cooling and heating required.

The difficulty experienced in the early stages of Run No. 10 in reducing the temperature gradient in the wax mass to the normal figure is reflected in the small yields of the lower-melting-point waxes. It is probable that the shock cooling promotes the formation of very small crystals—particularly in the surface region of the wax—which are not conducive to easy sweating, especially in the early stages.

SUMMARY.

The principal deductions from the fractional melting separation of waxes of different melting points from an oil-free wax stock may be summarized as follows :—

(1) The melting point–equilibrium temperature diagram obtained in the previously described[2] 14-stage equilibrium experiments is closely similar to that obtained at a low rate of sweating—3 per cent. per hour—in an experimental sweater. The data from the experimental sweater, worked at low rates, represent a nearer approach to the ideal infinite-stage process than that from the multi-stage equilibrium experiment. It is suggested that the diagram obtained at rates of 3 per cent. per hour or less in an experimental sweater, such as that described in this paper, be used as a basis for computing sweating efficiency.

(2) The rate of melting is a very important variable, and the efficiency of the process decreases with increase in the rate at which the process is conducted.

(3) Preparation of the sample by shock cooling to room temperature results in a wax mass which is more difficult to sweat during the early stages of the process.

(4) Cooling the sample to a temperature slightly below the complete solidification point, instead of to room temperature, before commencing the melting process results in a mass which is considerably easier to control during the early stages of the run. As the yields of the products are only very slightly lower than those obtained when the mass is cooled to room temperature, it may be concluded that the more extensive cooling is uneconomical, and therefore unnecessary.

(5) Local overheating, even to the extent of 2–3° F., has an adverse effect on the yields of the various waxes.

References.

[1] Sawyer, Hunter, and Nash, *J. Inst. Petrol.*, 1940, **26**, 390.
[2] *Idem, ibid.*, 1940, **26**, 430.

Department of Oil Engineering and Refining,
The University,
Birmingham.

THE IMPROVEMENT OF MIXED CRESOLS FOR INHIBITOR PURPOSES: THE USE OF MESITOL AS A GUM INHIBITOR.*

By T. Kennedy, M.Sc., Ph.D., A.I.C.

Summary.

Since the problem of improving mixed cresols as gum inhibitors consists in increasing the number of *ortho-* and *para-*alkyl substituents, a method for the synthesis of mesitol from commercial cresols has been investigated and shown to be possible, although experimental variables have not been worked out. It has been shown by direct comparison at various concentrations that mesitol has six times the efficiency of *o*-cresol as a gum inhibitor on a weight-to-weight basis.

Egloff, Morrell, Lowry and Dryer (*Industr. Engng Chem.*, 1932, **24**, 1375) have shown that *ortho-* and *para-*alkyl substitution increases the efficiency of a phenol as an inhibitor of gum formation in cracked spirits. The problem of improving the mixed cresols for inhibitor purposes is therefore the problem of increasing the number of *ortho* and *para* substituents in the cresol molecule. This may be carried out in a variety of ways. Thus B.P. 431,945 (1936, E.I. du Pont de Nemours) records the condensation of phenols with aldehydes in presence of sulphuric acid, whilst U.S. 2,058,881 (1937, Universal Oil Products) covers the product of reaction of olefins from cracked oil with a wood-tar distillate.

U.S. 2,061,111 (1937, Gulf Oil Co.) relates to the inhibitor produced by reacting cracked distillate with phenols in presence of sulphuric acid, and U.S. 2,149,759 (1939, Gulf Oil Co.) covers the use of the products of alkylation of phenols by refinery gases containing olefins in the presence of a small proportion of sulphuric acid.

It appeared to us that mesitol (2 : 4 : 6-trimethylphenol) would be a very efficient inhibitor, since it has both the *ortho* positions and the *para* position substituted by methyl radicals. Accordingly, mesitol was synthesized in the usual manner and its effect as an inhibitor of gum formation in Iranian cracked spirit compared with that of *o*-cresol by an accelerated oxidation test.† The results are shown in Fig. 1, from whence it is seen that mesitol has an efficiency of six times that of *o*-cresol on a weight-to-weight basis.

Although mesitol possesses many advantages over the mixed cresols, such as less solubility in water, and, being a colourless solid, is more easy to handle than the oily mixtures obtained by alkylation processes, it compares most unfavourably in price with the cresols, and a cheaper method of obtaining it was looked for. In the course of the investigation the following interesting observations were made and have been recently briefly reported in *Chemistry and Industry* (Kennedy, 1940, 59, 297). The method investigated was an adaptation of the method of Caldwell and

---

* Paper received 1st June, 1940.

† A modification of that of Hunn, Fischer and Blackwood (*S.A.E.J.*, 1930, **26**, 34), which will be described in a later paper.

Thompson (*J. Amer. chem. Soc.*, 1939, **61**, 765), who hydrogenated the *ortho*-dimethylaminomethyl derivative of *sym.*-metaxylenol prepared according to Decombe (*Compt. rend.*, 1933, **196**, 866) by the combined action of dimethylamine and formalin on the phenol. In the presence of copper chromite catalyst (Adkins, Conner, and Folkers, *J. Amer. chem. Soc.*, 1932, **54**, 1145) in dioxan solution at 165° C. and at a pressure of hydrogen of 2600 lb. a yield of 58·5 per cent. of 2 : 3 : 5-trimethylphenol was obtained.

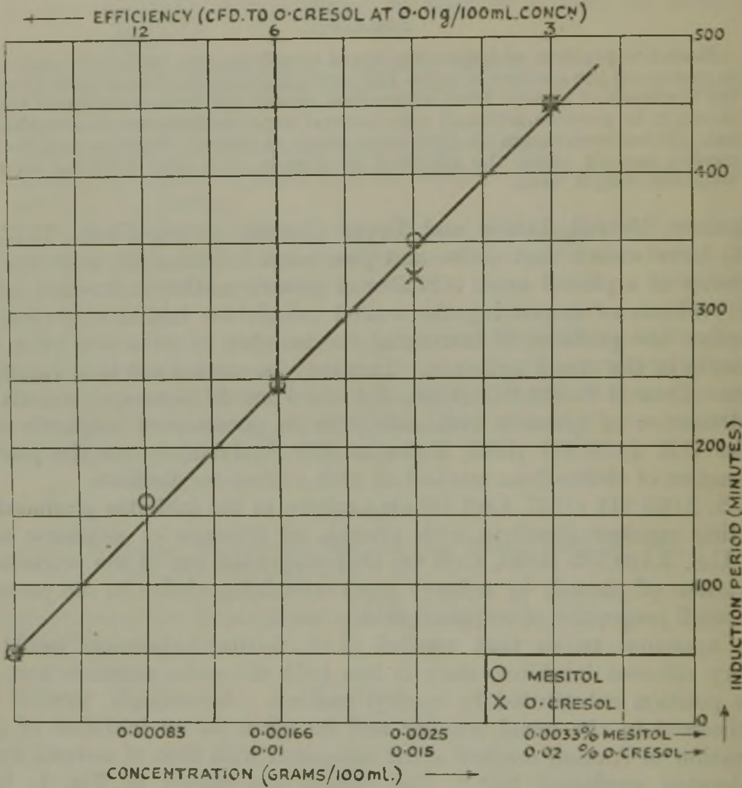


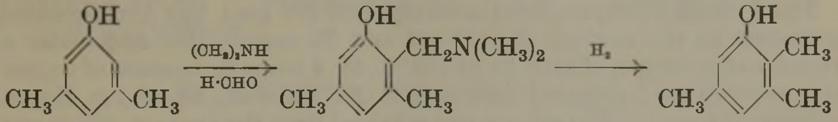
Fig. 1.

We have omitted the use of dimethylamine, and have used this reaction for the introduction of *two* methyl groups into the phenol molecule by the use of a compound which is obtained in almost theoretical yield from *p*-cresol, and which was first synthesized by Allmann and Buttner (*Ber.*, 1909, **42**, 2540) by the addition of two molecules of formaldehyde to an alkaline solution of the cresol.

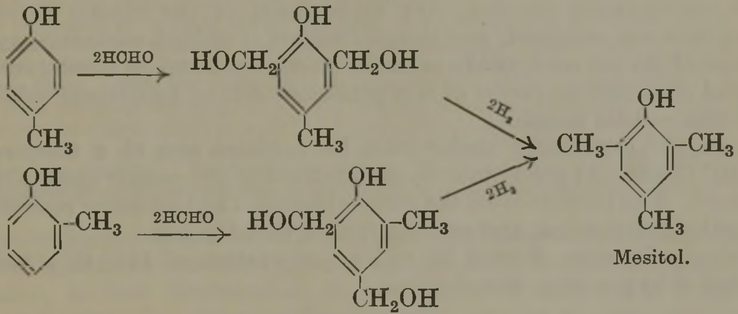
It was only possible to investigate the hydrogenation of this compound under very few conditions of temperature and pressure before the outbreak of war, but a yield of 25 per cent. of pure mesitol (m.pt. 70°) was obtained in 4 hours in presence of copper chromite in dioxan solution at a temperature of 170° C. and at a pressure of hydrogen of 2100 lb. Lowering of the temperature to 140° C. gave a yield of 10 per cent. of mesitol at 1900 lb.

pressure, whilst at a higher temperature (240° C.) and 2600 lb. pressure the benzene ring had apparently been hydrogenated, since no product soluble in caustic soda was obtained; the neutral residue had the very characteristic odour of methyl*cyclo*hexanol, and was undoubtedly mainly trimethyl*cyclo*hexanol.

Diagram I.



Caldwell and Thompson.



It was only possible to try one set of conditions for the mixed cresols. In this case the crude product from the reaction of two moles of formaldehyde and a commercial mixed cresol was hydrogenated at a temperature of 180° C. and a pressure of 1800 lb. A yield of 2 per cent. of mesitol was obtained. It is readily seen from Diagram I that both *ortho* and *para* cresols will give mesitol by this series of reactions, and there appears to be no reason to doubt that the latter compound could be obtained in quantitative yield from mixed cresols by this method, but the variables, pressure, temperature, type and volume of solvent, type and amount of catalyst all remain to be further investigated.

EXPERIMENTAL.

Addition of Formaldehyde to p-*Cresol.* 2 : 6-*Dihydroxymethyl*-4-*methylphenol.*

The method used was that of Allmann and Buttner (*loc. cit.*).

p-Cresol (108 gm. 1 mole) was dissolved in a solution of caustic soda (50 gm.) in water (200 c.c.) and an aqueous solution of formaldehyde (35 per cent., 215 c.c.) added. The mixture was left at room temperature for 4 days and the precipitated sodium salt filtered off. After another 4 days a further quantity was obtained, which was added to the first, and the whole acidified at 0° in an atmosphere of nitrogen by slow addition of a solution of acetic acid (10 per cent.). On warming, the mass dissolved, and on cooling, crystallized out in large colourless plates, which after further crystallizations from aqueous methanol, aqueous acetic acid, or ethyl acetate had m.pt. 130·5° (von Auwers, *Ber.*, 1907, **40**, 2531 gives

BIBLIOTEKA POLITECHNIKI

m.pt. 133°); yield 95 per cent. The dihydroxymethyl-*p*-cresol readily decolorizes in air, and prolonged boiling in solution yields insoluble polymerization products.

HYDROGENATION.

Mesitol.

The 2 : 6-dihydroxymethyl-4-methylphenol (10 gm.) was hydrogenated according to the method of Caldwell and Thompson (*loc. cit.*) under a pressure of hydrogen of 2100 lb. at 170° C. for 4 hours in presence of copper chromite (10 gm., prepared according to Adkins *et al.*, *loc. cit.*) in dioxan solution (200 c.c.). The solvent was removed from the product, which was dissolved in ether and the ethereal solution washed with water and extracted with caustic soda solution. On acidification of the caustic extract a precipitate was obtained, and steam distillation yielded colourless crystals of mesitol (25 per cent. yield), m.pt. 70°, both alone and on admixture with mesitol obtained by fusion of the potassium salt of mesitylene sulphonic acid with caustic potash.

A similar experiment under 2600 lb. pressure and at a temperature of 240° C. gave no precipitate on acidification of the caustic extract of the product. The product from the ethereal solution had an odour reminiscent of methyl*cyclo*hexanol, and was not further investigated.

Using a pressure of 1900 lb. and a temperature of 140° C. a yield of mesitol of 10 per cent. was obtained.

Production of Mesitol from Tricresol.

The addition of formaldehyde was effected as for *p*-cresol, but after a week the whole mixture was acidified and the crude, coloured mixture of unchanged cresols and addition products was hydrogenated as before, at 1800 lb. pressure and 180° C. On working up the product as described above, a yield of 2 per cent. of pure mesitol was obtained.

Inhibition Tests by the Accelerated Oxidation Test.

Solution under test.	Inhibition period (mins.).
Neat cracked spirit	50
,, ,, + 0·00083 gm./100 c.c. mesitol	160
,, ,, + 0·00166 gm./100 c.c. mesitol	245
,, ,, + 0·01 gm./100 c.c. *o*-cresol	245
,, ,, + 0·0025 gm./100 c.c. mesitol	350
,, ,, + 0·015 gm./100 c.c. *o*-cresol	325
,, ,, + 0·0033 gm./100 c.c. mesitol	450
,, ,, + 0·02 gm./100 c.c. *o*-cresol	450

Thanks are due to the Chairman of the Anglo-Iranian Oil Co., Ltd., for permission to publish these results.

Anglo-Iranian Oil Co., Ltd.,
Research Station,
*Sunbury-on-Thames.**

---

* Author's present address, c/o A.I.O.C., Abadan, Khuzistan, S. Iran.

THE EFFECT OF ALKYL SUBSTITUTION ON THE EFFICIENCY OF PHENOLS AS INHIBITORS OF GUM FORMATION IN PETROLEUM PRODUCTS.*

By T. Kennedy, M.Sc., Ph.D., A.I.C.

Summary.

The effect of alkyl substitution on the efficiency of phenols as inhibitors of gum formation in cracked spirits has been more thoroughly investigated in the case of the six xylenols, and observations have been made on the higher methyl substituted phenols. The conclusions of Egloff *et al.* (*Industr. Engng Chem.*, 1932, **24**, 1375) have been confirmed, but an "*ortho* effect" has been noted in the cases of 1 : 5 : 6-xylenol, durenol, and mesitol. Substitution by higher-alkyl groups has apparently no advantage over methyl substitution in the *para* position, although a slight advantage was observed in the *ortho* position. The molecular efficiencies (as compared to the cresols) of eighteen phenols are recorded for gum inhibition in Iranian cracked spirits.

The autoxidation of numerous natural and synthetic products on standing in air and light is an extensively occurring phenomenon of great economic importance, in that deterioration of commercial products often results by the production of gums, peroxides, and other unwanted products before use.

The problem of gum formation in petroleum products is generally held[1] to be due to the autoxidation of unsaturated hydrocarbons and the production of peroxides which may further decompose either at room temperature or on heating—*e.g.*, in an internal-combustion engine—to give gums. Before autoxidation commences, however, there is generally an induction period during which no peroxides are formed. Since petroleum products may have to be stored for a period before use, it is obviously advantageous to be able to increase this induction period by means of "inhibitors" of autoxidation.

The tendency of a product to form gum on standing may be assessed by noting the time taken by the product before appreciable oxidation occurs under standard conditions. A method in general use is that of Hunn, Fischer, and Blackwood[2] in which 200 c.c. of the oil are subjected to a pressure of 100 lb. per sq. in. of oxygen at a temperature of 100° C. in a bomb, and the time taken in minutes before a sharp drop in pressure is recorded, showing that the inhibition period is ended and that oxidation has set in, is taken as a measure of the efficiency of the inhibitor or of the potential gum-forming quality of the oil. Any comparison of efficiencies determined by this method will, of course, be a comparison at 100° C. and 100 lb. per sq. in. pressure of oxygen, and not at room temperature and pressure, but it is believed that this method does in fact give a good indication of the behaviour of the substance used as inhibitor under storage conditions. The efficiency of various gum inhibitors can be assessed by

---

* Paper received 1st June, 1940.

determining the "induction period" produced by a dilute solution of the inhibitor in the unstable liquid against a blank on an inhibitor of known efficiency.

Egloff, Morrell, Lowry, and Dryer [3] have studied the inhibitor action of a large number of substances on the autoxidation of a Pennsylvania cracked spirit in this manner, and found that modifying groups in amines and phenols were most effective in the *ortho* or *para* position, but that they exerted some beneficial influence when placed *meta* to the inhibiting group. They used a concentration of 0·01 gm. of inhibitor in 100 gm. of cracked spirit, and Table I gives the results they obtained on monohydric phenols.

Two alkyl groups were found to be more than twice as effective as a single group, and substitution in either *ortho* or *para* positions was considered to be nearly equivalent from a comparison of the four xylenols tested. They concluded that an *iso*propyl group *ortho* to the hydroxylic group is more effective than a methyl group, since thymol gave a longer induction period than carvacrol.

TABLE I.

Egloff et alii.[3]

Substance.	Induction period (min.).
Pennsylvania cracked spirit treated with fuller's earth	45
Phenol (0·01 gm. per 100 gm. spirit)	75
o-Cresol ,,	135
m-Cresol ,,	75
p-Cresol ,,	210
Butylphenol * ,,	195
tert.-Butylphenol * ,,	210
1 : 2 : 4-Xylenol ,,	420 §
1 : 2 : 6-Xylenol ,,	420 §
1 : 3 : 4-Xylenol ,,	255 §
1 : 2 : 5-Xylenol ,,	285
Thymol † ,,	300
Carvacrol ‡ ,,	180

* *ortho* and *para* mixed.
† 2-*iso*Propyl-5-methylphenol.
‡ 2-Methyl-3-*iso*propylphenol.
§ In benzene solution.

Because of the fact that the phenols isolated from petroleum fractions are often many times as efficient as the lower phenols, and since these "natural inhibitors" are in all probability simply mixtures of higher alkylphenols,[4] it was decided to investigate further the effect of alkyl substitution on inhibitor efficiency in the phenol series. The results reported below have been obtained in a similar manner to those of Egloff *et al.*, but using a smaller bomb (25 c.c. of cracked spirit). The bomb containing the sample in an open test-tube and connected to a recording pressure gauge was filled to a pressure of 100 lb. per sq. in. of oxygen and immersed in a steam bath. The steam was turned on and the time (T_1) noted. The pressure increased to a maximum due to increase in temperature, and remained steady for some time before dropping more or less sharply. The induction period was taken to be ($T_2 - T_1$), T_2 is the time at which the pressure has dropped to 2 lb. below the maximum pressure.

The spirit used was Iranian refined low-pressure cracked spirit, and a fresh sample was taken each week and standardized against *o*-cresol. When not in use it was kept sealed, in a refrigerator at 0° C. The characteristics of a typical sample of this cracked spirit are given in Table II. Little variation was shown from week to week.

TABLE II.

Specific gravity	0·764/60° F.
Elemental sulphur	0·0002%
Sulphur content	0·024%
Doctor test	Negative
Colour 18 in. cell (Lovibond) I.P.T. series	Water white (1·0)
C.F.R. engine test (Octane No.)	69·5
I.B.P. (A.S.T.M. Distillation)	43° C.
10%	75·5° C.
50%	130° C.
90%	166·5° C.
F.B.P.	183° C.

The procedure in each case was to determine the induction period for one phenol at various concentrations between 0·0005 and 0·05 gm. per 100 c.c.

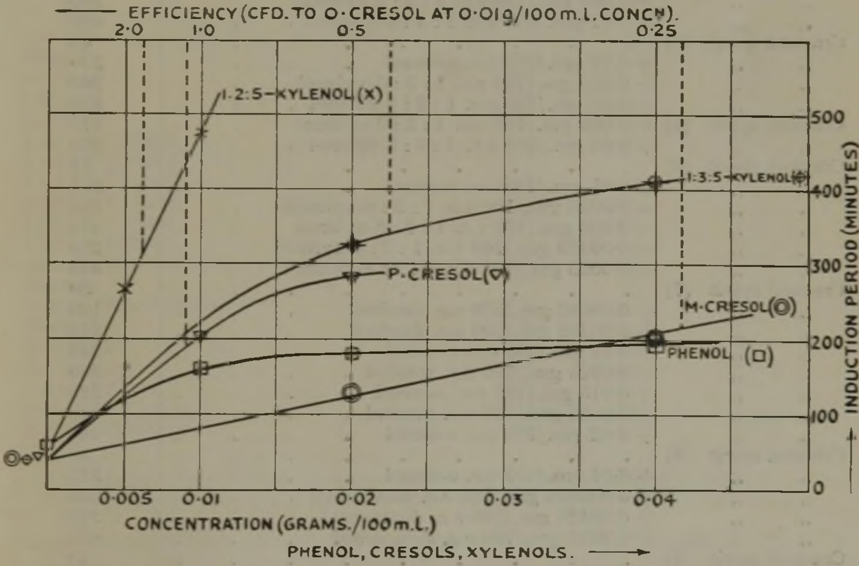


FIG. 1.

of cracked spirit, depending on the efficiency of the phenol. The induction period was plotted against the concentration of inhibitor and the concentration required to give an induction period equivalent to that given by 0·01 gm. of *o*-cresol read off. Then the efficiency of the phenol was taken to be 0·01 divided by the concentration read off from the graph. Molecular efficiencies were calculated by multiplying by the molecular weight of the phenol and dividing by the molecular weight of *o*-cresol. The results are given in Tables III and IV and are shown graphically in Figs. 1–7. There were two exceptions to the above procedure: *o*-amylphenol

TABLE III.

Substance.	Induction period (min.).
Cracked spirit (1)	60
,, ,, + 0·01 gm./100 c.c. *o*-cresol	230
,, ,, + 0·01 gm./100 c.c. phenol	160
,, ,, + 0·02 gm./100 c.c. phenol	180
,, ,, + 0·04 gm./100 c.c. phenol	195
Cracked spirit (2)	40
,, ,, + 0·01 gm./100 c.c. *o*-cresol	215
,, ,, + 0·02 gm./100 c.c. *m*-cresol	130
,, ,, + 0·04 gm./100 c.c. *m*-cresol	200
Cracked spirit (3)	45
,, ,, + 0·01 gm./100 c.c. *o*-cresol	190
,, ,, + 0·01 gm./100 c.c. *p*-cresol	205
,, ,, + 0·02 gm./100 c.c. *p*-cresol	280
Cracked spirit (4)	40
,, ,, + 0·01 gm./100 c.c. *o*-cresol	340
,, ,, + 0·02 gm./100 c.c. 1 : 3 : 5-xylenol	325
,, ,, + 0·04 gm./100 c.c. 1 : 3 : 5-xylenol	410
,, ,, + 0·005 gm./100 c.c. 1 : 3 : 4-xylenol	260
,, ,, + 0·01 gm./100 c.c. 1 : 3 : 4-xylenol	490
Cracked spirit (5)	40
,, ,, + 0·01 gm./100 c.c. *o*-cresol	310
,, ,, + 0·005 gm./100 c.c. 1 : 2 : 5-xylenol	265
,, ,, + 0·01 gm./100 c.c. 1 : 2 : 5-xylenol	470
Cracked spirit (2) + 0·005 gm./100 c.c. 1 : 2 : 3-xylenol	175
,, ,, + 0·01 gm./100 c.c. 1 : 2 : 3-xylenol	255
Cracked spirit (6)	55
,, ,, + 0·01 gm./100 c.c. *o*-cresol	355
,, ,, + 0·0025 gm./100 c.c. 1 : 2 : 6-xylenol	340
,, ,, + 0·005 gm./100 c.c. 1 : 2 : 6-xylenol	475
,, ,, + 0·00125 gm./100 c.c. 1 : 2 : 4-xylenol	270
,, ,, + 0·0025 gm./100 c.c. 1 : 2 : 4-xylenol	440
Cracked spirit (7)	50
,, ,, + 0·00083 gm./100 c.c. mesitol	160
,, ,, + 0·00166 gm./100 c.c. mesitol	245
,, ,, + 0·01 gm./100 c.c. *o*-cresol	245
,, ,, + 0·0025 gm./100 c.c. mesitol	350
,, ,, + 0·015 gm./100 c.c. *o*-cresol	325
,, ,, + 0·0033 gm./100 c.c. mesitol	450
,, ,, + 0·02 gm./100 c.c. *o*-cresol	450
Cracked spirit (8)	40
,, ,, + 0·01 gm./100 c.c. *o*-cresol	375
,, ,, + 0·000625 gm./100 c.c. *ψ*-cumenol	205
,, ,, + 0·00125 gm./100 c.c. *ψ*-cumenol	340
,, ,, + 0·0025 gm./100 c.c. *ψ*-cumenol	665
Cracked spirit (9)	50
,, ,, + 0·01 gm./100 c.c. *o*-cresol	355
,, ,, + 0·000625 gm./100 c.c. *unsym.*-hemimellitenol	170
,, ,, + 0·00125 gm./100 c.c. *unsym.*-hemimellitenol	245
,, ,, + 0·0025 gm./100 c.c. *unsym.*-hemimellitenol	415
,, ,, + 0·0025 gm./100 c.c. *sym.*-hemimellitenol	175
,, ,, + 0·00375 gm./100 c.c. *sym.*-hemimellitenol	285
,, ,, + 0·005 gm./100 c.c. *sym.*-hemimellitenol	405
Cracked spirit (10)	55
,, ,, + 0·01 gm./100 c.c. *o*-cresol	370
,, ,, + 0·00125 gm./100 c.c. durenol	250
,, ,, + 0·0025 gm./100 c.c. durenol	430
,, ,, + 0·000625 gm./100 c.c. prehnitenol	205
,, ,, + 0·00125 gm./100 c.c. prehnitenol	395

TABLE III (*continued*).

Substance.	Induction period (min.).
Cracked spirit (11)	45
,, ,, + 0·01 gm./100 c.c. *p*-cresol	220
,, ,, + 0·01 gm./100 c.c. *p*-*tert.*-butylphenol	200
,, ,, + 0·02 gm./100 c.c. *p*-*tert.*-butylphenol	290
,, ,, + 0·02 gm./100 c.c. *p*-*iso*-octylphenol	205
,, ,, + 0·04 gm./100 c.c. *p*-*iso*-octylphenol	430
Cracked spirit (12)	40
,, ,, + 0·02 gm./100 c.c. *o*-cresol	200
,, ,, + 0·02 gm./100 c.c. *o*-amylphenol	205
,, ,, + 0·04 gm./100 c.c. *o*-amylphenol	315

TABLE IV.

	Efficiency compared with *o*-cresol at 0·01 gm./100 c.c. concentration.	Molecular efficiency.
Phenol	—	—
m-Cresol	0·25	0·25
o-Cresol	1·0	1·0
p-Cresol	1·1	1·1
1 : 3 : 5-Xylenol	0·5	0·55
1 : 2 : 3-Xylenol	1·35	1·5
1 : 3 : 4-Xylenol	1·5	1·7
1 : 2 : 5-Xylenol	1·6	1·8
1 : 2 : 6-Xylenol	4·0	4·5
1 : 2 : 4-Xylenol	6·0	6·8
sym.-Hemimellitenol	2·1	2·6
Durenol	5·0	6·9
unsym.-Hemimellitenol	5·5	6·9
Mesitol	6·0	7·6
ψ-Cumenol	7·0	8·8
Prehnitenol	8·0	11·1
o-Amylphenol	1·0 *	1·5 *
p-*tert.*-Butylphenol	0·8 †	1·1 †
p-*iso*-Octylphenol	0·5 †	1·0 †

* Compared with *o*-cresol at concentration of *o*-cresol of 0·02 gm./100 c.c.
† Compared with *p*-cresol at concentration of *p*-cresol of 0·01 gm./100 c.c.

was compared with 0·02 gm. of *o*-cresol and *p*-(*tert.*-butyl)phenol and *p*-*iso*-octylphenol were compared with 0·01 gm. of *p*-cresol per 100 c.c. of cracked spirit.

Of the phenols used in this investigation the following were obtained from the sources shown :—

Phenol	40–41° B.P. detached crystalline.	May and Baker.
o-Cresol	" Pure."	Hopkins and Williams.
p-Cresol	,,	,, ,, ,,
m-Cresol	98–100%	British Drug Houses.
1 : 3 : 4-Xylenol	" Pure."	Gesel. für Teerverwertung.
1 : 2 : 4-Xylenol	,,	,, ,, ,,
1 : 2 : 5-Xylenol	,,	,, ,, ,,
1 : 3 : 5-Xylenol	,,	Monsanto Chemicals, Ltd.
p-*tert.*-Butylphenol	,,	,, ,, ,,
o-Amylphenol	,,	Sharples Solvent Corp., Philadelphia.

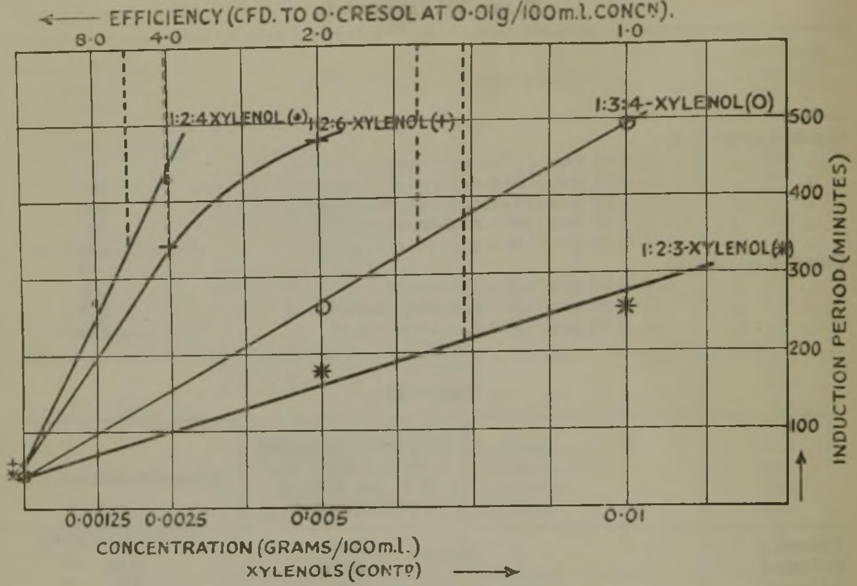

EFFICIENCY (CFD. TO O·CRESOL AT 0·01g/100m.l. CONCN).
8·0
4·0
2·0
1·0
1:2:4 XYLENOL (•)
1:2:6-XYLENOL (+)
1:3:4-XYLENOL (O)
1:2:3-XYLENOL (✳)
500
400
300
200
100
INDUCTION PERIOD (MINUTES)
0·00125
0·0025
0·005
0·01
CONCENTRATION (GRAMS/100 m.l.)
XYLENOLS (CONTD)

FIG. 2.

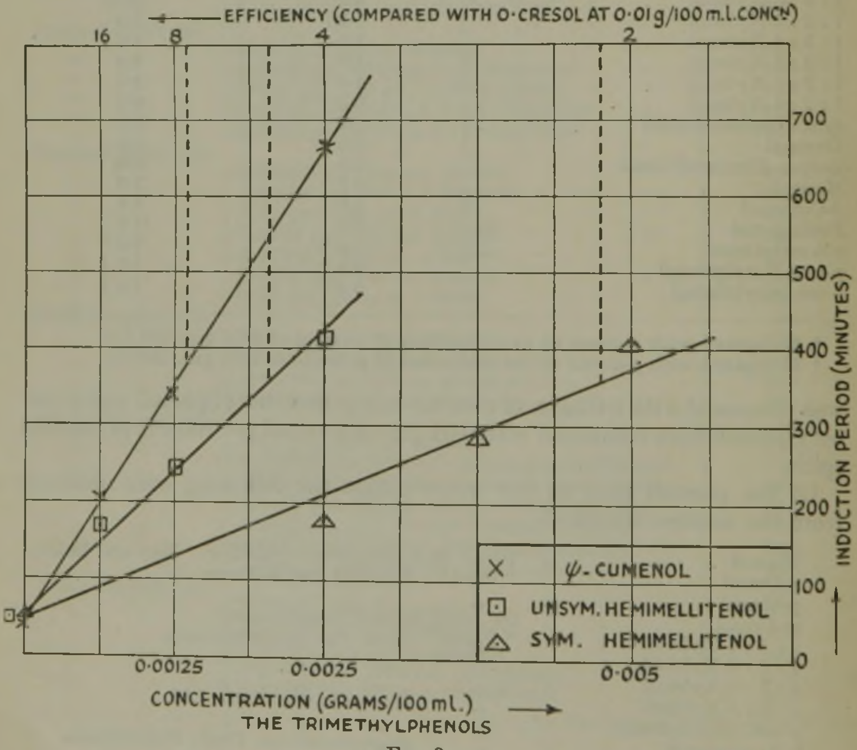

EFFICIENCY (COMPARED WITH O·CRESOL AT 0·01g/100 m.l. CONCN)
16
8
4
2
700
600
500
400
300
200
100
0
INDUCTION PERIOD (MINUTES)
× ψ-CUMENOL
▣ UNSYM. HEMIMELLITENOL
△ SYM. HEMIMELLITENOL
0·00125
0·0025
0·005
CONCENTRATION (GRAMS/100 ml.)
THE TRIMETHYLPHENOLS

FIG. 3.

1 : 2 : 6-Xylenol was obtained by Clemmensen reduction of 2-hydroxy-3-methylbenzaldehyde prepared from *o*-cresol by the method of Tiemann and Schotten.[5] The preparation of 1 : 2 : 3-xylenol presented much more difficulty. The method of Short, Stromberg, and Wiles,[6] a six-stage process starting by the nitration of *m*-cresol to 2-nitro-*m*-cresol and involving a Sandmeyer reaction on the corresponding amino-compound and a final Grignard stage, was thought to be unsuitable for the preparation of a small amount of this phenol, which was more readily prepared by the nitration of *o*-xylene,[7] reduction, separation of the 3-amino-*o*-xylene from the 4-amino-*o*-xylene by means of the formyl derivative,[8] and conversion of the amine by the action of nitrous acid into the corresponding 1 : 2 : 3-xylenol.

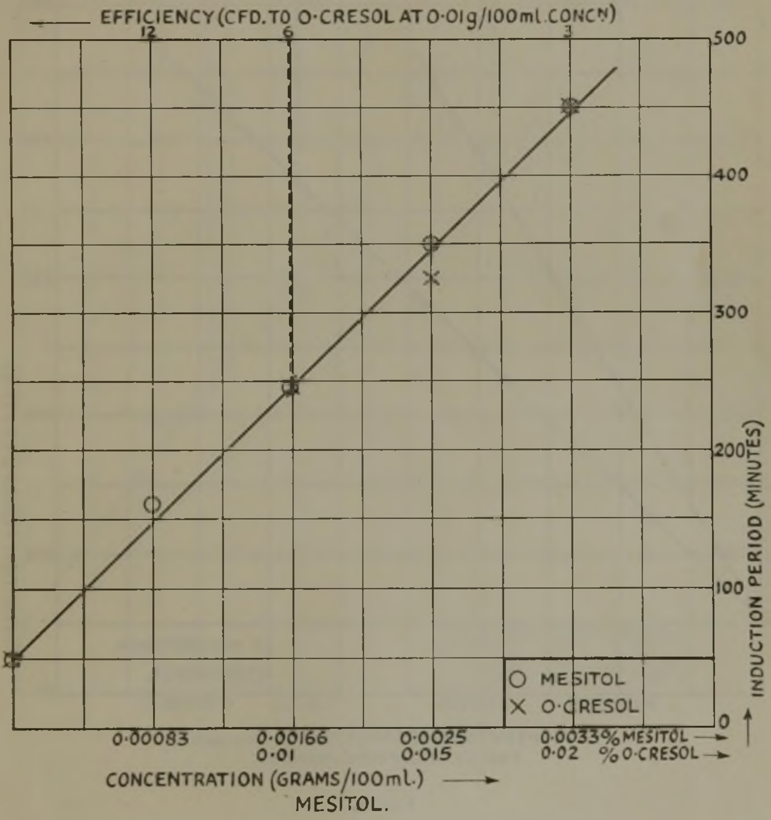


FIG. 4.

Mesitol, *ψ*-cumenol, *unsym.*-hemimellitenol, durenol, and prehnitenol were obtained by sulphonation of the corresponding hydrocarbons and fusion of the sodium salts of the sulphonic acids with caustic potash. Durene was prepared by the method of Smith [9] by methylation of xylenes. The residual tetramethylbenzenes after removal of durene were subjected to the Jacobsen rearrangement [10] and prehnitene isolated from the product. *sym.*-Hemimellitenol was obtained by Clemmensen reduction of 2 : 6-dimethyl-4-hydroxybenzaldehyde; the latter compound was isolated in good yield from 1 : 3 : 5-xylenol by Gatterman's method. p-*iso*-Octylphenol was prepared acording to Niederl [11] from phenol and di*iso*butene.

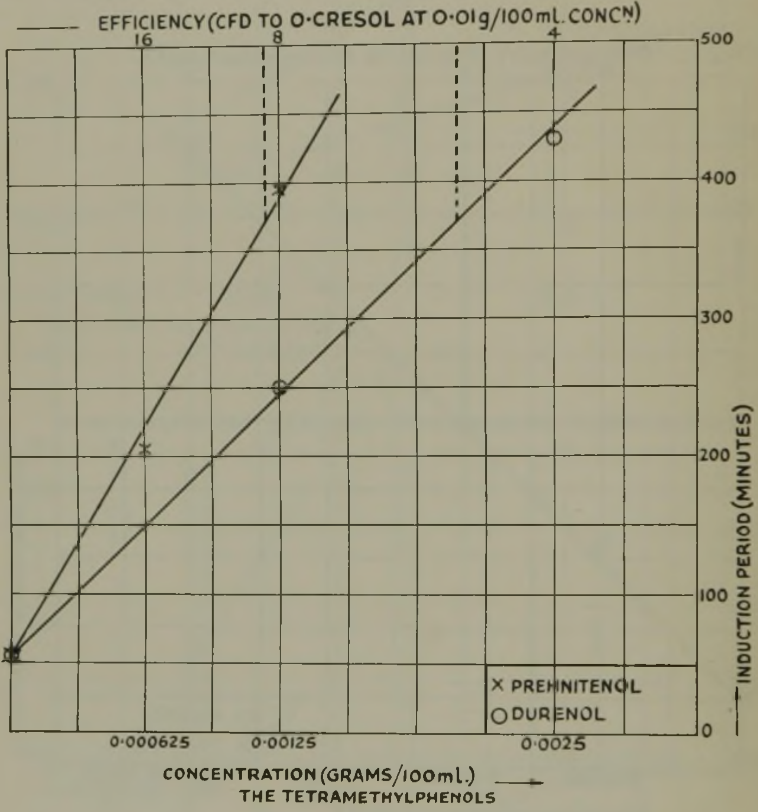


FIG. 5.

From the results certain conclusions may be reached. Thus since the efficiencies of *o*- and *p*-cresols are approximately the same and the efficiencies of 1 : 2 : 3-, 1 : 3 : 4-, and 1 : 2 : 5-xylenols are approximately the same, it is confirmed that the effects of *o*- and *p*-methyl groups are identical. It is also seen, as reported by Egloff, that when two methyl groups are present, the increase in efficiency is greater than would be expected from simple addition of the effects produced in the cresols. This is observed in 1 : 2 : 4-xylenol, where the combined effect of *o*- and *p*-substitution is much greater than the sum of the efficiencies of *o*- and *p*-cresol. This is shown to be also true of the tri- and tetra-methylphenols. Thus substitution in the *meta*-position by a third methyl group in 1 : 2 : 4-xylenol to produce *ψ*-cumenol confers a far greater increase in efficiency than would be expected by simple addition of the efficiencies of *m*-cresol and 1 : 2 : 4-xylenol. This effect is not so noticeable when a *meta*-methyl group is the second substituent. Thus 1 : 3 : 5-xylenol has approximately the efficiency expected by simple addition and 1 : 2 : 3-, 1 : 3 : 4-, and 1 : 2 : 5-xylenols are not very much more efficient than would be expected by adding the efficiencies of the appropriate cresols. No explanation is offered of the difference in efficiency of *unsym.*-hemimellitenol and *ψ*-cumenol. It is noted, however, that 1 : 2 : 3-xylenol is similarly slightly less efficient than 1 : 2 : 5-xylenol.

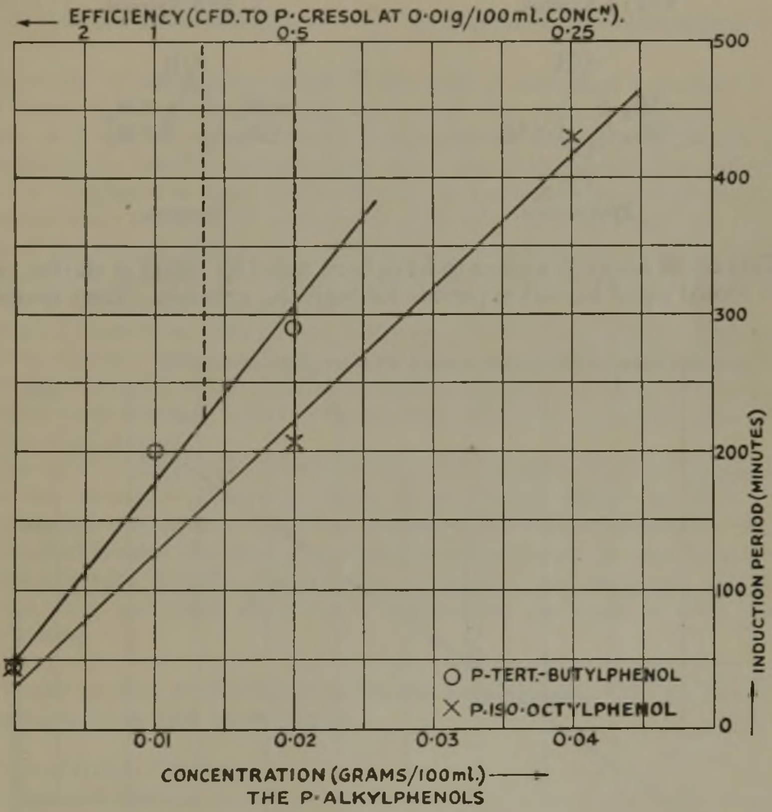
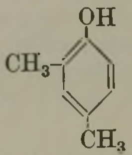


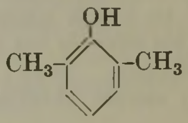
FIG. 6.

The cases of durenol and 1 : 2 : 6-xylenol require special notice. It has been seen that the effect of *ortho*- and *para*-substitution was approximately equal in the cresols and xylenols, but the above two phenols possess an appreciably less efficiency than would be expected on comparison with

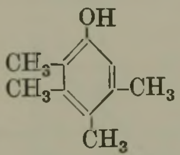
1: 2 : 4-xylenol and prehnitenol respectively. It is obvious that we are here dealing with a typical "*ortho* effect."



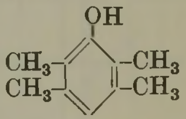
1 : 2 : 4-Xylenol.



1 : 2 : 6-Xylenol.



Prehnitenol.



Durenol.

This effect does not appear to have been noted by Egloff *et al.* (*loc. cit.*), who record equal induction periods for both the xylenols. They recorded

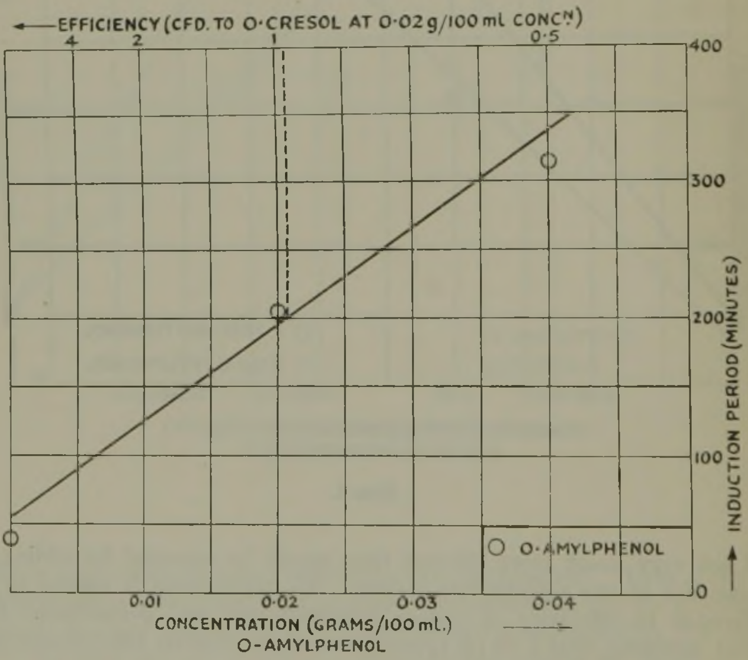
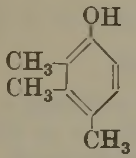
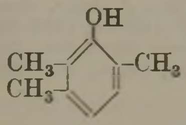


Fig. 7.

the results for one concentration only, however. The third case where this effect might have been noted is in the case of *unsym.*-hemimellitenol and

1 : 2 : 5-trimethyl-6-hydroxybenzene, but, owing to lack of time, it was not possible to synthesize the latter phenol.


unsym.-Hemimellitenol.


1 : 2 : 5-Trimethyl-6-hydroxybenzene.

It would be interesting to see if the critical oxidation potentials[12] of the above three pairs of phenols show any discrepancy due to an *ortho* effect, or if this effect is noticeable in the relative strengths of the phenols as reflected by rates of esterification, etc. The critical oxidation potential of 1 : 2 : 6-xylenol is cited by Egloff *et al.*, in a comparison of this property with inhibitor efficiency,[13] as equal to that of 1 : 2 : 4-xylenol, but in the case of the former phenol the potential is a calculated one, and obviously assumes the equality of *ortho-* and *para*-methyl substitution irrespective of the possibility of an *ortho* effect.

Mesitol is another example of a phenol possessing two *ortho* substituents, and its efficiency is not as great as might be expected from the fact that it contains two *ortho-* and one *para*-methyl groups. In fact, it is not as efficient as ψ-cumenol, which contains only one *ortho-*, one *para-*, and one *meta*-methyl group.

The type of alkyl substituent in the *para* position does not appear to alter the molecular efficiency, since the molecular efficiencies of *p-tert.*-butylphenol and *p-iso*-octylphenol are equal to that of *p*-cresol. From the limited results obtained on *o*-amylphenol, however, it does appear, in agreement with Egloff's observations on thymol and carvacrol (Table I above), that molecular efficiency is affected by the type of *ortho* alkyl substituent.

Thanks are due to the Chairman of the Anglo-Iranian Oil Co., Ltd., for permission to publish these results.

Anglo-Iranian Oil Co.,
Research Station,
*Sunbury-on-Thames.**

References.

[1] Article in " Science of Petroleum," Vol. II, p. 1016, by Gruse.
[2] *S.A.E.J.*, 1930, **26**, 34. See also Egloff, *et al.*[3]
[3] *Industr. Engng Chem.*, 1932, **24**, 1375.
[4] Tanaka and Kobayashi, *J. Fac. Eng. Tokyo Imp. Univ.*, 1927, **17**, 127–133; *Proc. Imp. Acad. Tokyo*, 1937, **3**, 595–596; Holzmann and von Pilat, *Brennst. Chemie*, 1930, **11**, 409–413.
[5] *Ber.*, **11**, 770.

---

* Author's present address, c/o A.I.O.C., Abadan, Khuzistan, S. Iran.

[6] *J. chem. Soc.*, 1936, 322.
[7] Crossley and Renouf, *ibid.*, 1909, 216.
[8] Hodgkinson and Limpach, *ibid.*, 1900, 68.
[9] *Organic Syntheses*, X, 32.
[10] Smith and Cass, *J. Amer. chem. Soc.*, 1932, **54**, 1621.
[11] *Industr. Engng Chem.*, 1938, **30**, 1269.
[12] Fieser, *J. Amer. chem. Soc.*, 1930, **52**, 5204.
[13] *Industr. Engng Chem.*, 1933, **25**, 804.

ABSTRACTS.

AUTHOR INDEX.

The numbers refer to the Abstract Number.

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

Geology and Development.

1.* Falls City Development Heralds Widespread Drilling in Basin. D. Dalrymple. *Oil Gas J.*, 31.10.40, **39** (25), 25.—After many dry holes in the Falls City basin, mainly in Missouri, the Falls City pool was found in Nebraska. A second oil pool has been discovered in Nebraska, an oil and gas pool in Kansas, and three gas pools in Missouri.

The cover of glacial drift has been one of the obstacles to development. Oil and gas have been found in the top of the porous siliceous dolomites of the upper Hunton and in the Misener sand just above. The Viola and the Bartlesville sand are also expected to yield oil.

Oil is obtained from the Hunton at depths of about 2200 ft. in the Falls City pool. There is a dome running north-west to south-east for 4 ml., and it is about 2 ml. wide. In the Shubert area oil comes from the Hunton at 2433 ft. The Mississippian is productive at 1594 ft. in one well at McLouth, Kansas. Showings of oil have been found in the Missouri sector of the basin, but few of the wells have been soundly located from a geological point of view.

A list of the present wildcats is given. There is also a geological column for the basin and a map showing the location of the pools. G. D. H.

2.* Future Oil Possibilities of Newhall–Castiac District. D. Wosk. *Oil Gas J.*, 17.10.40, **39** (23), 24.—The Newhall–Castiac district is one of the most promising in the Los Angeles region. It was productive for some years, and then lay dormant until 1937, when a field was discovered on a small monoclinal fold which is closed by an east-west fault on the south. The wells range up to 7350 ft. in depth, with an initial production of more than 5000 brl. from the Modelo.

In September 1940 a well blew in on the Del Valle anticline 3 ml. to the north-west. It gave 10–15 million cu. ft. of gas/day with 400–500 brl. of oil, from the Modelo at 6653–6900 ft.

The oil history of this region dates from 1856, and for many years oil was obtained from the upturned edges of the Modelo, from which there were many seepages. Drilling was chiefly near the crest of complex, steeply folded, and faulted structures. There were numerous small long-lived fields.

The beds in this area are of Miocene and younger age, with the marine Modelo giving the chief production, any of its sand bodies being potential oil producers. Lenses are probably of importance with regard to accumulation.

The 9-ml. Pico anticline is the main feature favourable to oil accumulation south of the Santa Clara river. Along the axis the beds are steep, but flatten rapidly on the flanks. Wells have had oil showings. G. D. H.

3.* Increased Drilling Activity is Seen for Mid-Continent. W. V. Howard. *Oil Gas J.*, 14.11.40, **39** (27), 57.—For the first ten months of 1940 drilling is running 15% above that for the corresponding period of 1939. Drilling in Kansas and north Texas has increased materially in view of the decline in the Illinois production.

Nearly two-thirds of the increase in drilling activity has been in Appalachian fields. In Ohio there has been a keen search for gas. A new deep sand-play is developing in eastern Kentucky. Drilling has fallen in Michigan, although new fields have been opened in Allegan, Arenac, and Gladwin counties. Encouraging showings have been noted in the Thumb district. Indiana activity has increased by 30%. In Illinois, Salem, Louden, and Centralia are practically drilled up, and there have been no comparable discoveries for two years. The Forest City basin is still mainly one of random wildcatting. In Kansas the new fields at 3000–4000 ft. are shallower than most recent discoveries.

In Oklahoma the Seminole district is still active with a number of extensions and discoveries. Oklahoma's most recent major discoveries are along the Nemaha Ridge. Activity in Arkansas has fallen off by 25% in 1940. North Louisiana has a 25% rise, due to developments at Olla. Many wildcats have been drilled in Mississippi, with disappointing results on the whole. However, some have had encouraging shows of oil. On the Gulf Coast of Louisiana many fields have been found in 1940, old ones extended, and deeper sands developed, but drilling on the Texas Gulf Coast is below the 1939 level. Texas Panhandle's drilling is 50% above the 1939 figure, and the Rocky Mountain drilling, too, is above the 1939 level.

In 1940 there have been few discoveries in California. Declining production in the Los Angeles basin will encourage wildcatting, especially as a number of tests have been successful, as at Athens, Inglewood, and East Coyote. G. D. H.

4.* Finding of Deeper Pay Revives Iowa Field in South Louisiana. N. Williams. *Oil Gas J.*, 14.11.40, **39** (27), 82.—After being almost inactive for several years, new wells are being drilled and old ones deepened in the Iowa field. Formerly the deepest and main producing horizon was the 6900–7000 ft. Marginula sand, discovered in 1931. Recently a new horizon was found in the Lower Frio, just below 8200 ft. This sand is probably more than 40 ft. thick. It gave 633 brl. of oil in 24 hr. on a ¼-in. by 6-in. positive choke. The casing pressure was 1750 lb./in.^{2}, and the G.O.R. 737. The gravity of the oil is 36·2° A.P.I.

The 6900–7000 ft. sand is nearing exhaustion, and water has encroached, so that only 16 out of 49 wells are in production. Some of the watered wells have been plugged back to produce from shallower sands. About 35,000,000 brl. of oil have been obtained from the 6900–7000 ft. sand, chiefly under natural flow. The sand has an active water-drive, and water encroachment has been gradual and almost uniform. The field is domal, and is thought to overlie a deep-seated salt dome.

The main shallower sands are at 4400–4500 ft., 5000–5200 ft., 5500 ft., and 6600–6700 ft., and they also have active water-drives. Except for the 6600–6700-ft. sand they are all in the Miocene.

The new deep sand seems as though it may be the most prolific, for it is thicker and may be more extensive than the shallower sands. G. D. H.

5.* New Pool in Prospect in Gallatin County, Illinois. W. F. Hoover. *Oil Gas J.*, 14.11.40, **39** (27), 87.—In July 1939 a small well was brought in in the Cypress at 2421–2462 ft., after being drilled to the Fredonia and plugged back. Other wells were drilled in this area, but, with the exception of one which gave 175 brl. in six months, all were dry. In September 1940 a 200–300-brl. well was completed in the Rosiclare member of the Ste. Genevieve, and is believed to point to the existence of an oil-pool. Data are given regarding the wells drilled in the area.

The structure is probably pre-Chester, with about 50 ft. of closure. There may be a north-east–south-west fault downthrown to the north-west.

The potential productive acreage is 600–800 acres. G. D. H.

6.* Claim Panhandle and Hugoton Gas Reserves Ample to Supply Wisconsin Markets. Anon. *Oil Gas J.*, 14.10.40, **39** (27), 88.—The gas reserves at Hugoton, Kansas, are estimated at 18,000,000,000,000 cu. ft., with the present annual withdrawals about 30,000,000,000 cu. ft. The Hugoton field covers 2,980,000 acres, and there are 330 wells averaging 2800 ft. in depth. The reservoir pressure is 409–430 lb./in.^{2}

The Panhandle field is said to cover 1,475,000 acres and to have 22,195,000,000,000 cu. ft. This field has 1652 gas wells of an average depth of 3000 ft. The present reservoir pressure is about 340 lb./in.^{2} G. D. H.

7.* Well Logs and Field Data of Active Oil Areas—South Central Oklahoma. Anon. *Oil Gas J.*, 14.11.40, **39** (27), 122.—The main development began in 1913 with the discovery of the Healdton field. There oil is in Upper Pennsylvanian and Ordovician sands and limes. More than a score of fields have been developed in the west of Carter County. Madill gave some oil from the Trinity (Cretaceous).

Structurally the area is very complicated, with steep dips. The trends were mainly determined in pre-Pennsylvanian times, and the Pennsylvanian is highly irregular. Oil is mainly in irregular Pennsylvanian sands and the Viola and Simpson of the Ordovician. G. D. H.

8.* California Dry Gas Reserves. L. P. Stockman. *Oil Gas J.*, 14.11.40, **39** (27), 253.—Although dry gas fields are not yet of economic importance in California, they are likely to be so in the future. Dry gas is produced at the McDonald Island field, San Joaquin Co., and the Buttonwillow field, Kern Co. The dry gas reserve is of the order of a million million cubic feet.

The largest gas producer in the state is the Kettleman North Dome field, and even now it gives 40% of California's total gas output. The Rio Vista dry-gas field will probably yield 750,000,000,000 cu. ft. It is on an anticline which was discovered geophysically, and some 15,000 acres are said to be proved. The anticline trends N. 50° W., and its closure is apparently less than 50 ft. The Tulare is met at 4045 ft., and consists of sands, sandy clays, silts, and claystones. From 4045 to 5050 ft. are the San Joaquin clays; 5505–9200 ft. Etchegoin sands and Jacalitos; 9200–10,000 ft. Upper Miocene hard shales. The Miocene sand zone is at 10,000–11,000 ft. Some of the sands are oil-saturated, but failed to give production.

The Delano gas field is a dome discovered geophysically, with production in the top of the San Joaquin clay series. There are two zones ranging from 25 to 100 ft. in thickness. The productive capacity is 35,000,000 cu. ft./day.

The gas of the Chowchilla field contains 56% of nitrogen. In the Willows area a well was brought in with a production of 5,356,000 cu. ft./day. Commercial gas has been found 14 ml. west of Marysville, production being from the Cretaceous at 5855 ft.

California's gas-fields are listed, together with the relevant data. G. D. H.

Geophysics.

9.* Prospecting Principles. E. E. Rosaire. *International Oil*, July 1940.—Although prospecting has naturally always formed an integral part of petroleum industry, it is only recently that it has assumed a place comparable in importance with other branches, such as marketing, refining, transportation, and production. The author of this paper traces the development of prospecting and notes three " revolutionary " advances which have been made, the term " revolutionary " being used in a military sense to describe events which have drastically changed not only the conduct of a particular campaign, but equally the whole course of history.

Firstly there was the incidence of geological prospecting from which was evolved the anticlinal theory, forming a platform for all structural prospecting during the last 25 years. Next came geophysical prospecting, which enabled the prospector to explore below the surface of the earth by means other than the drill. Finally a new science of prospecting made its début, the principles of which are based on recognition and measurement of geochemical manifestations of petroleum accumulations, the trap within which such deposits occur being of secondary importance to the actual petroleum itself. In other words, structure, according to this technique of geochemical prospecting, is no longer regarded as a sufficient condition for petroleum accumulation.

The question is then asked as to how modern prospectors should proceed. Are there no basic principles recognized as characteristic of successful experiences? There is no doubt that there are, but unfortunately available evidence seems scarcely favourable to their easy understanding. The author has therefore endeavoured to deduce the fundamentals of prospecting by appeal to a broader field of endeavour, and has accentuated the principles of prospecting by analogy with the principles of war.

The United States army field regulations recognize nine principles of war as follows :

(1) The principle of the objective.
(2) The principle of the offensive.
(3) The principle of mass.
(4) The principle of economy of force.
(5) The principle of movement.
(6) The principle of surprise.
(7) The principle of security.
(8) The principle of simplicity.
(9) The principle of co-operation.

Although certain adjustments in viewpoint are necessary in order to translate these principles of war into principles of prospecting, it is possible to apply them to petroleum exploration. Thus the author suggests that the prospecting chief of staff, his superiors and his subordinates might well discard rule-of-thumb methods in favour of definite principles of prospection such as he has outlined. H. B. M.

10.* Geochemistry in Prospecting for Petroleum. E. E. Rosaire. *International Oil*, July 1940.—The author of this paper accentuates the importance of recognizing a petroleum accumulation as a dynamic entity rather than a static condition. Visualized thus, it is not difficult to trace the origin of significant hydrocarbons, such as ethane, propane, and butane, in near-surface soil. Even though the actual petroleum deposit is buried under tons of water-logged sediments, proximate rock pressures and concentration gradients are continuously forcing component hydrocarbons into and through its sedimentary environment.

Such continuous leakage over a protracted period results in chemical and physicochemical modifications of the surrounding and overlying sediments. The essence of geochemical prospecting lies firstly in detecting these manifestations of a petroleum accumulation, and secondly in their proper interpretation.

As a rule structural evidences of petroleum accumulation manifest themselves over or around the centre of the deposit. In the case of geochemical manifestations, however, these reach their maximum intensity around the edges of the petroleum accumulation. In other words, significant hydrocarbons which have escaped into surrounding or overlying sediments in the manner described form a halo or annular pattern in the soil. Thus it follows that soil analysis for the significant saturated hydrocarbons and detection of the halo is destined to play a vital part in geochemical prospecting technique.

Examples of the successful application of this method of petroleum exploration are already to hand. The importance of these achievements, however, lies not so much in the actual discoveries made, but in the knowledge that petroleum accumulations in stratigraphic traps are potentially capable of discovery by means other than the drill.

There is another aspect of geochemical prospecting which may have far-reaching results, and that is geochemical well-logging. Its successful application has already prevented the abandonment of wells as " dry " on the grounds that contract depth had been reached and none of the usual indications of petroleum possibilities were present. The geochemical well-log should, therefore, be carefully assessed before any decision is taken to abandon a hole. If it is favourable, it is more than likely that drilling to a greater depth will transform what appears on other evidences to be a " dry " well into a discovery well. H. B. M.

Drilling.

11.* Oil-Base Drilling Mud. Anon. *Petrol. World*, October 1940, **37** (10), 18.—In developing an oil-base mud it was found that, if properly proportioned, the following materials made a suitable rotary drilling fluid : (1) stove oil, to act as the liquid medium ; (2) oyster shells, limestone, or barytes, to serve as weighting materials, and also to impart the initial plastering properties ; (3) lamp-black, to give gel strength and structure ; (4) blown asphalt, to impart the final plastering properties.

The proportions normally used are 1 part blown asphalt mixed with 1½–2 parts of oil (by weight).

Normally oil-base muds require very little attention while in use in a well. The occasional small adjustments required can be effected by the addition of the proper materials. However, if any major adjustments are necessary, care should be taken to preserve or re-establish the proper inter-relationship between the ingredients.

Precautions should be taken to prevent contamination of the mud with water or water-base mud. Small quantities of water or clay do not affect the physical properties ; however, when large quantities of water (+ 15%) are present, the quality of the mud is apt to be seriously impaired.

When oil-base muds are subjected to temperatures above 200° F. for a period of 4 or 5 hrs. their physical properties are damaged. At present neither the reason nor the cure for this condition is known.

Oil-base muds have very little tendency to gas cut ; consequently it is permissible to maintain higher viscosities in the muds, to minimize the chances of settling in the bottom of the well when circulation is suspended. A. H. N.

12*. Chemicals for Treatment of Drilling Mud. C. F. Bonnet and B. N. Howell. *Petrol. World*, October 1940, **37** (10), 67.—Complex phosphates and tannic-acid compounds (tannin extracts) are widely and effectively used for controlling viscosity, gel strength, and water loss in drilling muds. By employing these chemicals low-cost fresh clays, hole-made muds, and muds contaminated in use can be improved and the drilling mud kept in good condition while wells are being drilled.

Complex phosphates quickly reduce viscosity and gel strength and lower water loss to a certain extent.

Tannin extracts reduce water loss and improve the quality of mud cake, sometimes reducing viscosity.

Sodium silicate reduces gel strength and under certain conditions lowers viscosity and water loss.

Sodium bicarbonate restores cement-cut mud.

Excessive treatment or repeated treatment with the same chemical for long periods is harmful. New testing methods make it possible to anticipate over-treatment and avoid difficulties.

While the characteristics of chemicals used in drilling mud are known, the mud is continually changing during use. For this reason frequent tests should be made to guide the treatment, and chemicals should be added on the recommendation of engineers familiar with drilling muds and chemicals.

The paper discusses the properties and uses of the chemicals themselves, and not the way these chemicals are actually used to meet field conditions. A. H. N.

13*. Advancements in California Drilling Practices in 1940. A. H. Bell. *Oil Gas J.*, 24.10.40, **39** (24), 30–32. *Presented before Petroleum division of American Institute of Mining and Metallurgical Engineers.*—Since the discovery of the extremely deep fields in the San Joaquin valley there has been a definite trend towards the development of large and heavy equipment to drill to deepest levels at high speed. This development reached its peak during the past year with the introduction of the 500-lb. steam-pressure superdrilling rig.

The development of extra-heavy drilling equipment for great depths has naturally been followed by higher depreciation costs on this more expensive equipment, and the operators have been forced to drill at greater speeds, even against the handicaps of greater depths, in order to reduce overall drilling costs.

The greatest saving in the slim-hole programme is through reduced size of casing, the water-string usually being 5 in. in diameter. One operator is experimenting with the use of 4½-in. o.d. tubing for drill-pipe and then cementing the tubing in for casing, thus doing away with drill-pipe investment and maintenance. The requirement of the California State Division of Oil and Gas for protection strings cemented over upper oil zones offers some difficulty in planning reduced well costs. Many operators have lately been seeking modification of these rules, claiming that cementing and mudding practices have been greatly improved since the rules were laid down in earlier years, and that the upper zones can now be effectively protected by mudding and cementing behind one water-string.

Cementing practices, mud control, and weight indicator design are briefly discussed. A. H. N.

14*. Unusual Two-Zone Completion Method Used in South Texas. E. C. Will. *Oil Gas J.*, 24.10.40, **39** (24), 35–36.—The usual method of completing a well in two sands is to set a tubing-to-casing packer between the two producing horizons and allow the lower sand to produce through the tubing and the upper sand to produce through the annular space between the casing and tubing. This method, however, was not always practical, for reasons which were discussed in detail. This method has also the great disadvantage of inability to start production from the upper sand flowing again should it cease. Production from the lower sand can always be maintained through the tubing by pumping, swabbing, or any of the other usual methods. However, it is impossible to swab, pump, or otherwise agitate the fluid in the annular space between the tubing and casing.

A new method is described, which incorporates a special patented bottom-hole valve. Two wells have been completed in this manner to date, and a third is now in process. The two completed wells have been producing without trouble for some 2½ months, and it is thought that this method shows considerable promise not only in solving problems of a similar nature in other areas, but also in solving difficulties which at first glance appear to be entirely unrelated. For example, with this device it will be possible to pump a well from two separate sands selectively. Also, in fields where gas lift is practicable, this device may be employed to great advantage in producing from two separate sands at will, by injecting gas through the tubing-string.
A. H. N.

15*. Effects of Different Properties of Rotary Drilling Fluids. H. F. Simons. *Oil Gas J.*, 31.10.40, **39** (25), 46–47.—The principal features of the mud which demand the attention of the engineers, superintendents, and drillers are : (1) weight ; (2) viscosity ; (3) gel strength ; (4) water loss ; (5) wall-building properties ; (6) thixotropy ; (7) acidity or alkalinity, and (8) salinity. In this paper only the first two properties in the list are studied.

A factor not to be overlooked in the study of mud weight necessary to prevent blowouts is that the hole must be kept full of fluid if the pressure is to be maintained. As the drill-pipe is withdrawn, the fluid in the hole recedes, and unless the hole is kept filled, the point may be reached where the formation pressure exceeds the fluid pressure ; the results are the same as occur with failure to use a sufficiently heavy mud. Air or gas in the mud will lighten it, and gas-cut mud will sometimes have a specific gravity less than 1, due to the expansion of the entrained gas as it rises to the surface and the pressure is reduced.

By increasing the viscosity, the flow of the fluid may be changed from the turbulent to the plastic type, and the efficiency with which the cuttings are removed is affected accordingly. Some consider that the flow of the fluid in a drilling well is of the plastic type, whilst others think it approaches the turbulent type, due to the velocity of the fluid, the effect of the motion of the drill-pipe, and the slippage of the cuttings.

For viscous flow the slippage of the cuttings is computed by the formulæ : $v = 1{\cdot}789d^2(f - f_2)$ for round cuttings and $v = 0{\cdot}696d^2(f_1 - f_2)$ for flat cuttings.

For turbulent flow the equations for slippage are : $v = 9\frac{d(f_1 - f_2)}{f_2}$ for round cuttings and $v = 3{\cdot}5\frac{d(f_1 - f_2)}{f_2}$ for flat cuttings, where v = slip in ft./sec. ; d = diameter of round or major diameter of flat cuttings ; f_1 = density of cutting in lb./cu. ft. ; f_2 = density of mud in lb./cu. ft.
A. H. N.

16.* Continuous Logging at Rotary Drilling Wells. J. T. Hayward. *Oil Gas J.*, 14.11.40, **39** (27), 100. *Paper presented before American Petroleum Institute.*—The author discusses the development of procedure and equipment now being used successfully in detecting relatively small quantities of oil, water, and gas brought to the surface from the bottom of a drilling well by mud-circulating fluid used in rotary drilling, together with the method used in determining the depths of the horizons where the samples entered the well-bore.

He describes in detail a number of instruments that have been placed on a panel and installed in a trailer ; the trailer is moved from location to location as the drilling rig is moved. Most of these instruments are concerned with the logging of the well as drilling progresses. With these new instruments and methods it is now possible to drill ahead continuously, and to know fairly accurately the type of formation that is being drilled through—its oil, water, and gas contents, and its depth. Information of this nature, obtainable while drilling, is not only helpful in determining completion procedures, but may also become useful in the later life of the well in producing or reconditioning operations.

The trailers and service have now been commercially available a little more than a year, and have been used on approximately 200 wells. In addition to logging, the trailers are at present equipped for routine drilling-mud control and for core analysis. The development is in the early stages, and it is expected that other functions connected with the logging and operations of drilling wells will be added as time goes on. A. H. N.

17.* Hydraulics of Mud-circulating Systems. W. W. Moore. *Oil Gas J.*, 14.11.40, **39** (27), 208. *Paper presented before American Petroleum Institute.*—This discussion does not relate to mud control nor mud conditioning, about which much has already been published. It deals rather with a preliminary analysis of the problem of circulating an average rotary-drilling mud down to and back up from the bottom of the well-bore in such volume and at such velocity as effectively to remove drill cuttings from the face of the formation being contacted by the drill bit and to transport such cuttings up to the surface, where they can be deposited.

The useful work accomplished by the circulating mud stream is normally very small in relation to the energy expended. Much of the hydraulic energy furnished by the slush pump is consumed by friction head within the drill-pipe; whereas no useful work is accomplished by the mud stream until after it has left the drill-bit. In most instances the overall mechanical efficiency of the mud-circulating system can be improved by closer attention to the relationship of hole diameter, drill-pipe size, and slush-pump capacity. Included with this paper are charts and tables which it is believed may assist in analysing the problem introduced here.

It is concluded that: (1) Results in practice, so far as they have been observed, seem to confirm the theoretical advantage of keeping the ratio of drill-pipe size to hole diameter as high as possible, while retaining adequate clearance for fishing operations. (2) Drilling costs are influenced to some extent by the size, weight, and initial cost of the drilling equipment required for a specific operation. More efficient use of slush-pump capacity, therefore, should result in lowered drilling costs. (3) The subject, here discussed qualitatively, would seem to be of sufficient importance to warrant further and more scientific study, so that a quantitative analysis of the various factors involved could be made available to the industry. A. H. N.

18.* Errors in Acid-Bottle Readings. G. H. Scott. *J. Inst. Petrol.*, 1940, **26** (201), 349–356.—A chart of correction, based on experimental data, is given by means of which acid-bottle readings may be corrected to give values accurate enough for all practical purposes. A. H. N.

19. Patents on Drilling. J. C. Curtis. U.S.P. 2,217,674, 15.10.40. Appl. 5.5.38. Portable drill-rig on a wheel-supported carriage.

D. Scaramucci. U.S.P. 2,217,708, 15.10.40. Appl. 8.5.39. Well-cementing method and apparatus which includes as a step the introduction of an exothermic substance ahead of the cement.

R. K. Henderson. U.S.P. 2,217,747, 15.10.40. Appl. 9.6.39. Drillable packer.

J. M. Pearson. U.S.P. 2,217,768, 15.10.40. Appl. 5.11.37. Apparatus for orienting cores by a suspended magnetic system.

R. W. Lyon. U.S.P. 2,217,803, 15.10.40. Appl. 18.11.39. Swivel for drilling oil wells with lubricated inner and outer sets of discs for supporting the hollow stem.

H. T. Byck. U.S.P. 2,217,857, 15.10.40. Appl. 17.4.37. Process for the removal of mud-sheaths by means of an electrode, insulated from the well and maintaining a high positive potential with regard to the mud-sheath.

M. B. Willey. U.S.P. 2,217,879, 15.10.40. Appl. 16.1.40. Method and apparatus for drilling into water-covered ground using submersible barge and foundation, which are later removed to new locations.

C. E. Carpenter and R. C. Lynch. U.S.P. 2,217,889, 15.10.40. Appl. 20.10.38. Diamond drill-bit with a concave working face and a water-way.

P. V. Campen. U.S.P. 2,217,926, 15.10.40. Appl. 23.8.37. Non-aqueous drilling mud with a peptizing agent consisting of an organic nitrogen base.

P. V. Campen. U.S.P. 2,217,927, 15.10.40. Appl. 23.8.37. Non-aqueous drilling mud containing a substance which is soluble in the oil-base and having a sulphur-containing acid radical.

A. J. Penick and K. T. Penick. U.S.P. 2,218,093, 15.10.40. Appl. 9.12.37. Blow-out preventer with a movable ram.

W. W. Gipson, F. N. Fossati, and R. H. Long. U.S.P. 2,218,131, 15.10.40. Appl. 10.2.39. Fishing-tool for wells using a low-voltage transformer to make a flash weld with a metallic object.

P. M. Rea. U.S.P. 2,218,267, 15.10.40. Appl. 31.7.39. Fishing-tool of the overshot type.

K. Nishio. U.S.P. 2,218,295, 15.10.40. Appl. 7.11.38. Apparatus for obtaining core from rotary core-boring.

L. V. Mallory. U.S.P. 2,218,337, 15.10.40. Appl. 8.11.37. Apparatus for recovering implements from wells.

H. W. Ball. U.S.P. 2,218,412, 15.10.40. Appl. 3.10.38. Sheave-block having antifriction bearings.

B. S. Minor. U.S.P. 2,218,473, 15.10.40. Appl. 18.11.39. Swivel with lubricated supporting members.

J. G. Phillips. U.S.P. 2,218,496, 15.10.40. Appl. 16.1.39. Take-off tool for wells.

E. E. Huebotter. U.S.P. 2,218,533, 22.10.40. Appl. 6.2.39. Method and apparatus for filtering and dehydrating drilling mud.

H. L. Strader. U.S.P. 2,218,728, 22.10.40. Appl. 5.4.40. Cementing hook with a flexible cement-conducting member connecting hook and casing-head closure.

D. L. Butler. U.S.P. 2,218,742, 22.10.40. Appl. 22.3.38. Cathead construction.

A. C. Catland. U.S.P. 2,218,743, 22.10.40. Appl. 23.4.37. Well-reamer comprising a cutter on a pin and ball bearings.

L. P. Parker. U.S.P. 2,218,766, 22.10.40. Appl. 22.6.40. Pipe-cutting tool the knives of which are actuated by a piston which is responsive to mud pressure after a valve is closed.

A. D. Maclachlan. U.S.P. 2,219,047, 22.10.40. Appl. 12.1.39. Hose and coupling structure.

D. Miller. U.S.P. 2,219,110, 22.10.40. Appl. 15.8.38. Mast for drilling oil wells and the like.

G. W. Bowen. U.S.P. 2,219,148, 22.10.40. Appl. 3.1.39. Inside pipe-cutter with a hydraulic control for the downward movement of tool and feed of the cutter to the work.

F. N. Osman. U.S.P. 2,219,151, 22.10.40. Appl. 17.3.38. Cable-socket using a soft moulded metal around cable.

P. Hirsch. U.S.P. 2,219,266, 22.10.40. Appl. 9.11.39. Hose- and pipe-coupling consisting of a screw-connected socket and nipple engaging the end of the hose.

O. N. Maness. U.S.P. 2,219,325, 29.10.40. Appl. 9.1.39. Method of cementing wells, the seal being outside a perforated liner and using a non-penetrating liquid to drive the sealing liquid through the liner.

E. Wiemer. U.S.P. 2,219,435, 29.10.40. Appl. 28.8.37. Process and device for treating muds.

A. T. Cooper and L. F. Valentine. U.S.P. 2,219,512, 29.10.40. Appl. 21.3.38. Device for surveying drilled holes by means of a plumb-bob and compass which are locked to show their position inside the well-bore.

W. Wright. U.S.P. 2,219,880, 29.10.40. Appl. 10.8.38. Pipe-clamp.

D. V. Waters. U.S.P. 2,220,983, 12.11.40. Appl. 3.6.39. Apparatus for power-flow control utilizing two power-supply members, one at constant speed, driving a differential gearing system.

L. F. Brauer. U.S.P. 2,220,989, 12.11.40. Appl. 14.4.39. Well clean-out bailer with a special type of valve at bottom to allow variation in size of debris which can be forced through an aperture by fluid pressure.

J. H. Wilson. U.S.P. 2,221,067, 12.11.40. Appl. 19.2.38. Structural steel derrick which is made of two halves, which are raised by means of a central mast.

J. J. Santiago. U.S.P. 2,221,204, 12.11.40. Appl. 25.4.38. Cementing plug adapted to be lowered on a suspension pipe and to deliver fluid from the pipe either below or above the packer.

D. McClelland. U.S.P. 2,221,242, 12.11.40. Appl. 28.1.39. Drill.

M. C. Nesbitt. U.S.P. 2,221,359, 12.11.40. Appl. 23.6.37. Hoist with two drums and special differential gearing which allows both drums to be used simultaneously from one source of power.

J. C. Fortune and W. R. Walne. U.S.P. 2,221,376, 12.11.40. Appl. 6.6.38. Rotary drilling equipment using a round flush-joint grief stem and slips to hold it firmly.

C. F. Baker. U.S.P. 2,221,392, 12.11.40. Appl. 14.12.38. Core-catcher.

A. R. Maier. U.S.P. 2,221,486, 12.11.40. Appl. 29.12.38. Guard for rotary machines. A. H. N.

Production.

20.* Pressure Maintenance. O. W. Willcox. *World Petrol.*, October 1940, **11** (10), 36–41.—The history and significance of pressure maintenance projects are reviewed. " Critical phenomena " and retrograde condensation and distillation and their importance in high-pressure pools are studied with particular emphasis on the gains to be expected from pressure maintenance in such areas. As an example the following case is cited :—

Studies of the history of the Kettleman field in California and Oklahoma City have resulted in the conclusion that these large petroleum deposits were originally under pressure and temperature such that a considerable proportion of their contents were in the sub-critical gaseous state ; that is, they were combination oil and distillate pools, with an upper gas-zone above a liquid oil-zone. It is said that if the pressure on these two fields had been maintained by cycling, vast amounts of natural gasoline, and even motor fuel, might have been obtained. But in those days the operators of those fields knew nothing about critical states and recycling operations ; the only use they had for a compressor was to pump what dry gas they could to consuming centres ; the rest could be thrown away. The consequence was that their formation pressures declined to a point where the valuable light hydrocarbons came out of the sub-critical state (retrograde condensation) and mingled with the general fluid contents of the pools. Superficially considered, this might not be supposed to represent a loss, as the liquid may still be subject to recovery by pumping or by an eventual repressuring. However, a hydrocarbon that exists in the sand in the sub-critical state is mobile and is 90% or more recoverable by rational pressure maintenance. On the other hand, even under the very best conditions scarcely 50% or 40% of the liquid contents of an oil reservoir are recoverable by any generally available means ; the rest wets the sand grains and sticks. A. H. N.

21.* Modern Well Studies. Part 3. F. Briggs. *Oil Wkly*, 21.10.40, **99** (7), 29.—Dynamometer cards obtained on the polished rod of a pumping well reflect loads composed of many factors, and for that reason are to a certain extent unpredictable. The loads set up in a sucker-rod string during the pumping motion are the ultimate combined results of (1) the weight of the rods, (2) the force required to accelerate the rods, (3) the weight of the oil, (4) the force required to accelerate the oil on the upstroke, (5) friction of the rod-string, (6) friction in the stuffing-box, (7) friction resulting from the motion of the plunger, (8) friction of the oil in the tubing, (9) a friction loss through the valves, and (10) stress waves and reflections contained within the rod-string which are created by any of the above impressed forces. These factors are not always taken into consideration; nor is it necessary to understand their significance and implications in normal dynamometer studies.

Vibration effects are studied in some detail. Sometimes vibrations in rod-strings are desirable, at other times they are of no consequence, and at still others they are troublesome and not wanted.

Well-pound is explained and illustrated in great detail. Over-travel and under-travel causes and effects are further studied and illustrated, and wells of the agitators, semi-agitators, and true pumpers are typified. An analogy to the electrical-power-factor term has been offered to describe the power or work of a pumping well. The term " mechanical power factor " may be aptly applied to this phase of a pumping well. In electrical terminology, power is said to be the result of the voltage multiplied by the amperes times the cosine of the phase angle of lag or lead of the two previous terms. In other words, power is equal to $EI \cos \theta$. Similar to this, the mechanical power factor may be the force times the velocity times the cosine of angle of lag between these two factors. It has been said that " if the force and velocity are cyclic in nature, they can be resolved into harmonic components. If these components are in phase, it is obvious, for a given power requirement and a given velocity, the force will be a minimum. If they are out of phase, the force must necessarily be larger to give the same power."

The paper is generously illustrated by many cards and graphs. However, the promise given of deriving Rieniet's equation in Part 3 does not materialize, although many of the variables considered in the formula are studied. (See Abstract No. 1557, 1940.)

A. H. N.

22.* Subsurface Waters Gaining Favour for Flooding. Anon. *Oil Wkly*, 21.10.40, **99** (7), 22–26.—The problem of water for flood projects differs in some important respects from demand for other oil-field operation. Once a project is started, the water supply must be available and sufficient to carry on the flood without interruption. Volumes may vary from 100 to 50,000 brls. daily. It must be free of matter that might clog the pores of the sand. It must not chemically react with formation water or strata compounds to precipitate a floc. In many respects the water must be more nearly pure than ordinary city waters. The treated supply must not corrode equipment unduly, nor must the over-all water-control cost be above a few cents at the input well.

Use of any type of surface supply leaves much to be desired. There is always the question of shortage during seasons of minimum rainfall. Flood stages are doubly bad; first, because there is danger of the dam breaking, second, because flood-waters increase dirt and silt content. Combined with these drawbacks is the necessity of changing the treatment to counteract the changes in the make-up water.

Lime strata as a water-source are then discussed in detail in so far as they fulfil the requirements of flooding from all these viewpoints. Treatment of the lime-water to render it safe and economical to use is detailed step by step, and the chemical reactions are explained where necessary.

It is concluded that the successful use of lime-water has provided the flood operations with a wider scope than has been possible hitherto. Some possible areas, previously condemned for immediate development through lack of surface-waters or uneconomic pipe-line distances, may now be developed, with the water being obtained from lime horizons at no increase in the per-barrel cost of the injected water.

A. H. N.

23.* Modern Well Studies. Part 4. F. Briggs. *Oil Wkly*, 28.10.40, **99** (8), 26.—This part forms the conclusion of the series of papers on well studies, and deals with the use of the well studies in counterbalancing wells.

Counterbalancing a single well is studied first. No study is made of the counterbalancing systems; only the magnitude and direction of the counterbalance are determined. The methods of slipping the clutch, using a tachometer, and of electric-power measurements are briefly discussed, and dynamometer card studies for counterbalance are dealt with in more detail. Typical third- and second-order cards are shown for good, over-, and under-counterbalance conditions. Changes effected in the shapes of the cards by improving the counterbalance in various typical cases are illustrated and the significance of such changes is discussed.

Finally, central-power counterbalancing is analysed. First, the procedure is given, which may be summarized as follows: Dynamometer cards are obtained on each of the rod-lines near the central power. The rod-lines are identified with the eccentric on which they work and the direction from which they leave the central power. The dynamometer cards are transferred to polar diagrams. The loads on the various rod-lines are determined from the dynamometer cards for each 30° interval of crank rotation. Vector diagrams of these forces or loads are drawn for each 30° interval of crank rotation. Resultants of each of these forces diagrams are resolved into the right angle and parallel component with respect to the crank position. Using the right-angle component, diagrams are drawn plotting these components against the angle of crank rotation. The horizontal components are then plotted against the angle of crank rotation. These resulting diagrams show the condition of the power with respect to torque and overturning force, from which weight changes can be made to give better operations.

Second, this procedure is illustrated by working out an example in complete detail, the example being an actual case of a central power for eleven wells operated from two eccentrics centred on one power-wheel, the eccentrics being one below, and 180° out of phase of the other.

[Cf. Abstracts Nos. 1557, 1559, (1940); and **21** (1941).] A. H. N.

24.* Pressure Project Involves 150 Individual Operators. Anon. *Oil Wkly*, 28.10.40, **99** (8), 19–24.—The largest co-operative pressure maintenance programme in the history of the oil business is being conducted in the K-M-A field of North Texas. This project involves co-operation among nearly 150 individual operators, owning more than 1300 wells and something like 25,000 producing acres.

The chief operating difficulty encountered has been that of hydrates in the gas, which froze connections and instruments at the point of greatest pressure drop between the distribution lines and the input well-head. When the gas was measured back to the leases for the pressure maintenance, it was found to be so " soft " that a great amount of research and investigation was necessary to establish a factor acceptable to those concerned for converting the visible meter readings to actual cubic feet. For the time being, a super-compressibility factor of 1·33 is being used.

To reduce the ice-forming propensity of the gas, most of the gasoline compression plants have facilities for drying. Activated alumina is used in two of the units, and two employ low-temperature refrigeration coupled with diethylene glycol for the removal of water. The plants which employ activated alumina have vessels installed so that they can be operated in units of one or more, and so that units of one or more can be regenerated by passing gas at a temperature between 350° and 400° F. through the material to drive out the absorbed moisture removed from the gas. The plants equipped with low-temperature chilling by expanding propane in specially designed heat-exchangers are the first of their kind to use diethylene glycol in direct contact with the commodity coupled with low-temperature chilling, and have been successful in reducing the dew point of the gas to 0° F., at 1800 lb. pressure. The plants and method of utilizing diethylene glycol are studied in general. A. H. N.

25.* Unusual Testing Methods Used in Texas Recycling Project. G. Weber. *Oil Gas J.*, 31.10.10, **39** (25), 48.—To facilitate control and testing of the distillate-producing wells, all producing wells in a particular field have been equipped with surface connections arranged to permit sampling and flow measurements. The well-head connections include a vertical loop in the line from the christmas tree. Accurate metering of gas is possible in the vertical meter setting, as no weep-holes are required for drainage of liquid through the orifice plate. The orifice meter is equipped with liquid seal-pots, which are kept half filled with condensate during warm weather.

This condensate may be replaced with anti-freeze solution such as ethylene glycol during the winter.

Samples of the well-fluid are obtained from a special connection on the upstream side of the vertical loop. The well-fluid at this point contains some distillate in the liquid phase, so it is necessary to use a special sampling device that will obtain a representative sample of the gas–liquid mixture leaving the well. For this purpose a special sampling tube is welded into the line. A sharp-edged, stream-lined nozzle of small diameter is attached to the lower end of the sampling tube, and is located in the centre of the flow-pipe, with the sharp open edge of the nozzle directed against the direction of fluid flow. A valve and threaded connection on the top of the sample tube permits connection of the sampling to the portable test unit. High-pressure flexible tubing is used for making this connection. Sampling procedure is detailed.

It is emphasized that the test procedure does not affect the producing well in any way. The rate at which the well is producing is not affected, and the back pressure on the well is not changed. Therefore, the well is tested under conditions that are entirely normal, and the question of the possibility of effect of abnormal flow conditions on the distillate content of the well-fluid, that are required in some suggested test procedures, is entirely eliminated. A. H. N.

26.* Application of Core Analyses to Production Methods. J. A. Lewis. *Oil Gas J.*, 31.10.40, **39** (25), 56.—The production of gas, oil, and water from a well takes place only after flow of these fluids through permeable reservoir formations. The analysis of cores has developed to a point where it is possible through accurate and complete analysis of contaminated or uncontaminated cores to predict with a high degree of accuracy the type of fluid production that will result by exposing a given cored interval to a pressure lower than that present in the formation. Having obtained gas or oil production, the problem resolves itself into one of evaluating the formation in terms of rate and ultimate production.

The discussion presupposes that the formation is oil productive and that accepted core-analysis methods were combined with connate water and residual oil saturation data in computations for recovery. These determinations, correlations, and applications in accordance with principles previously published by Muskat and Wyckoff were successfully made and applied in the water-flooding area of Pennsylvania.

It is expected that from a correlation of these formation and fluid characteristics with field operation a sound basis for oil-reservoir development and operation will result. A. H. N.

27.* Economics of Controlling Water Produced with Oil. J. U. Teague. *Oil Gas J.*, 14.11.40, **39** (27), 125. *Paper presented before American Petroleum Institute.*—The economics of doing remedial work to exclude the water from wells producing water and oil is a problem of serious concern to the operator.

The production of water in some fields is no doubt injurious to the oil-producing reservoir, and should be excluded to conserve reservoir energy and increase ultimate recovery. In many other fields there is no apparent damage to the reservoir by the production of water, and any remedial work done must be justified by the saving in operating cost resulting from not having to lift the water and then dispose of it.

The principal factors that should be considered in determining the economics of remedial work are : (1) physical characteristics of the reservoir ; (2) effect of water production on reservoir energy and ultimate recovery ; (3) means of determining the source of water ; (4) types of remedial work in common use ; (5) increased operating costs due to production of water with oil ; (6) disposal of water produced.

It is important that the statistical data and information necessary to evaluate these factors be accumulated prior to the time the production of water becomes acute and a decision on remedial work must be made. A. H. N.

28.* Oil Recovery by Air-Drive in Venango Fields of Pennsylvania. P. A. Dickey and R. B. Bossler. *Oil Gas J.*, 14.11.40, **39** (27), 161. *Paper presented before American Petroleum Institute.*—The secondary recovery of oil by air-drive for approximately 15 years has been practised generally in the Venango oil-fields of North-western Pennsylvania. Hitherto no attempt has been made to analyse the results and estimate the quantity of oil recoverable by this method.

The production-decline curves of properties on which no new development has taken place over a period of several years approximate straight lines on logarithmic paper. Following accepted procedure, the economic life and cumulative ultimate oil recovery of the properties may thus be predicted. Unfortunately predictions could be made for only a few of the older properties and for none of the newer developments, because on most leases new work so affected the oil production that no conclusions could be reached. Twelve leases showed suitable curves. The estimates of cumulative ultimate recovery range from 11 to 100 bbl./acre-foot, with an average of 42 bbl. The economic life ranges from 9 to 48 years, with an average of 28 years.

Whereas a large number of factors affects the recovery of oil by air-drive, it is believed that the principal ones are the original oil and water content of the sand, the spacing of the wells, and the efficiency of the application of air to the sand. The latter factor is seriously affected by the by-passing of air through the more permeable beds, and is difficult to take into account.

It is suggested that ultimate oil recovery and well spacing are connected by an equation of the type : $Y = ae^{-b \times P}$. Tentative values, based on the available data, were assigned to the constants. A. H. N.

29.* Factors Affecting Reservoir Performance. R. D. Wyckoff. *Oil Gas J.*, 14.11.40, **39** (27), 188.—It is pointed out that in attempting to outline concisely the factors affecting reservoir performance and to describe the technique necessary to determine these factors, the author is handicapped by the relatively immature state of development in reservoir technology. Important basic theoretical facts are known, and idealized analyses are possible. What is needed are the qualifying factors obtainable only from experience, which will permit the application of theory to practice.

The problems encountered are discussed under two main classifications : (1) factors inherent in the reservoir, and over which little or no control is possible ; (2) factors influenced by the exploitation programme (*i.e.* under partial or complete control).

The types of reservoirs are studied as affected by structural, pay-zone, and fluid-content characteristics. These characteristics belong to the first of the two classifications, and to determine them all possible information should be gathered. A discussion of core analysis and bottom-hole sampling and pressure measurements indicates the methods used to study and interpret these characteristics.

The factors influenced by exploitation programme present even greater complexities, because of interdependence of all these factors. The functions of a well are outlined and an analysis of location of wells in the reservoir and of well-diameter effects on production follow conventional lines.

Reservoir mechanics are discussed under water, gas, and combination drives, both natural and artificial. The differences exhibited by reservoirs due to the different types of drives or controls are outlined in generalities.

The study of well-spacing at the end of this comprehensive paper is of interest, as a discussion is made of the assumptions regarding radii of drainage of wells. It is stated that 10 years' intensive search for some physical phenomena in the production mechanism leading to a limited radius of drainage has failed to disclose its existence—not only in water or gas-cap drives but even in internal gas drives. Thus it is concluded that what really exists is not a physically but an economically limited radius of drainage.

The paper is in the form of a stimulating discussion and is free from dogmas.
A. H. N.

30.* Some Notes on Plunger Lift Operation. F. R. Wellings. *J. Inst. Petrol.*, 1940, **26** (195), 19–28.—The paper deals with actual experiences of wells on one field only, but as the wells considered possess such widely differing characteristics, it is thought the general principles so derived may be applicable to other wells. It is concluded that up to the time of writing the disadvantages of two-piece plungers outweigh the advantages. A. H. N.

31. Patents on Production. W. A. Fry. U.S.P. 2,217,676, 15.10.40. Appl. 27.5.37. Treatment of wells having calcareous formations with HCl having a calcium precipitating agent soluble in the acid solution.

J. B. Hawley, Jr. U.S.P. 2,217,746, 15.10.40. Appl. 22.2.37. Submersible pump with a shaft and an impeller.

E. V. Hewitt. U.S.P. 2,217,749, 15.10.40. Appl. 26.1.39. Liquid recovery and gas recycle method utilizing two wells simultaneously to produce from two formations having both gaseous and liquid hydrocarbons.

G. S. Knox. U.S.P. 2,217,986, 15.10.40. Appl. 29.4.40. Well-pipe plug adapted to be lowered and set in the well-casing.

J. B. Hawley, Jr. U.S.P. 2,218,003, 15.10.40. Appl. 25.2.37. Submersible pump motor.

A. D. Garrison and C. F. Teichmann. U.S.P. 2,218,025, 15.10.40. Appl. 10.6.38. Method of treating a well by setting a filter pack of a granular oil-wettable, water-repellent material with interstitial spaces of capillary dimensions.

G. W. Rusler and V. V. Vacquier. U.S.P. 2,218,155, 15.10.40. Appl. 17.1.40. Formation tester with a packer and check-valves.

C. M. O'Leary. U.S.P. 2,218,214, 15.10.40. Appl. 18.3.38. Pumping apparatus with balancing arrangement utilizing an incompressible fluid passing from one cylinder into another cushioning cylinder, the latter containing a compressible fluid.

C. M. O'Leary. U.S.P. 2,218,215, 15.10.40. Appl. 17.2.40. Air balance for pumps comprising a balancing cylinder closed at its opposite ends and a reciprocating piston forming upper and lower compression spaces.

C. M. O'Leary. U.S.P. 2,218,216, 15.10.40. Appl. 25.3.40. Fluid counterbalance for pumping apparatus for wells where walking-beams are used.

K. Austerman. U.S.P. 2,218,306, 15.10.40. Appl. 3.6.38. Method of treating oil-wells with an alloy of an alkali metal, the alloy having a specific gravity in excess of 1.

I. J. McCullough. U.S.P. 2,218,802, 22.10.40. Appl. 27.2.39. Gun perforator in which the products of explosion from the firing charge of one chamber is conducted to fire other charges in the succeeding chambers, the products being restrained from being lost.

N. H. Armstrong and R. M. Marr. U.S.P. 2,218,839, 25.10.40. Appl. 5.8.38. Stuffing-box for pumping well-casing.

J. W. Johnson. U.S.P. 2,218,955, 22.10.40. Appl. 24.2.39. Guide for flexible well lines when being lowered through well-casing.

M. O. Johnston and D. B. Chapman. U.S.P. 2,218,988, 22.10.40. Appl. 5.1.39. Adjustable flow-bean consisting of a series of ports in a tubular element, which ports can be successively opened or closed to increase or decrease rate of flow in the tubular element.

I. S. Salnikov. U.S.P. 2,219,022, 22.10.40. Appl. 25.7.39. Method for completing oil-wells in a formation of multiple producing zones whereby the diameter of the casing set in the formation is reduced against the pay-zones, the reduced sections being drilled and reamed off after the cement is set.

M. Jacobs. U.S.P.P. 2,219,161 and 2,219,162, 22.10.40. Appl. 28.11.38 and 21.8.39, respectively. Pipe couplings.

V. A. Hoover. U.S.P. 2,219,318, 29.10.40. Appl. 24.3.38. Motor construction for submersible purposes.

C. R. Irons. U.S.P. 2,219,319, 29.10.40. Appl. 18.5.38. Treatment of wells for sealing a formation by a liquid sealing agent and using a non-penetrating liquid above the sealing liquid to drive the sealing agent into the formation.

R. L. Ralston. U.S.P. 2,219,635, 29.10.40. Appl. 20.4.39. Device for eliminating gas-lock in pumps by means of a by-pass.

J. H. Notley. U.S.P. 2,221,057, 12.11.40. Appl. 18.7.39. Well agitator by means of a chamber which is alternately filled by, and emptied of, the well fluid.

M. Hector. U.S.P. 2,221,137, 12.11.40. Appl. 16.2.40. Safety cleaning-tool having a knife for cleaning deposits from oil-well tubing, pipes, casings, or formation.

P. J. Lehnhard, Jr. U.S.P. 2,221,261, 12.11.40. Appl. 2.5.39. Method of treating wells by forcing fluid into the formation through granular material located in the well, opposite the formation, and using a non-penetrating fluid to force the sealing fluid.

D. A. Limerick. U.S.P. 2,221,353, 12.11.40. Appl. 27.3.39. Process for treating an oil-well to clean it from organic deposits, using an exothermic reaction between acids to melt the deposits.

M. M. Kinley. U.S.P. 2,221,427, 12.11.40. Appl. 29.3.37. Swab, consisting of two swabs and a valve.

J. R. Mahan and J. B. Picard. U.S.P. 2,221,428, 12.11.40. Appl. 29.12.39. Counterbalance device consisting of weights carried on supports which are mounted on a shaft.
A. H. N.

Cracking.

32.* Solubility of Cracked Residues in Some Solvents. J. Voskuil. *J. Inst. Petrol.*, 1940, **26** (196), 57–71.—Experiments are reported on the solubility of cracked residues in aliphatic hydrocarbons as well as in *cyclo*hexane. The solubility of these residues in pure benzene is made a special study. The colloid–chemical nature of the cracked residue is demonstrated by comparison of extraction and dilution methods.
A. H. N.

Synthetic Products.

33.* Synthesis of Lubricating Oils by Condensation Reactions. Part 1. W. R. Wiggins, T. G. Hunter, and A. W. Nash. *J. Inst. Petrol.*, 1940, **26** (197), 129–145.—The study is that of the condensation of chlorinated paraffin wax with aromatic hydrocarbons, using metallic aluminium as catalyst to give viscous hydrocarbon oils of high viscosity indices and of viscosity–gravity constants comparable to those of paraffin base-oils.
A. H. N.

34.* Synthesis of Lubricating Oils by Condensation Reactions. Part 2. W. R. Wiggins, T. G. Hunter, and A. W. Nash. *J. Inst. Petrol.*, 1940, **26** (199), 225–234.—The engine-wear, oil consumption, carbon deposits, and conditions of oil in crank-case were determined for 2 synthetic oils. The oils were condensation products of chlorinated paraffin wax and benzene using metallic aluminium as catalysts in one case, and aluminium chloride in the second.
A. H. N.

Refining.

35. Refining Characteristics of Illinois Basin Crude Oils. G. Egloff, G. B. Zimmerman, and J. C. Morrell. *Oil Gas J.*, 26.10.39, **38** (24), 81.—The properties and yields on laboratory fractionation and cracking and qualities of products from Salem, Illinois Pipe Line, Loudon, Mount Carmel, and Sandoval crudes from the Illinois basin are given.

Salem crude yields on distillation 35·7% vol. of 400° E.Pt. gasoline of 53·5 O.N. and 25·6% of furnace-oil distillate. Combined topping and cracking increased the yield to 62·7% of 64 O.N. gasoline with 14·3% of No. 2 furnace distillate of pour point − 35° F. and 19·7% of residual fuel of Grade 6 quality with a viscosity of 222 secs. S.F. at 122° F. and a cold test of 45° F. The use of a catalytic polymer plant and an absorption plant would increase the yield of gasoline to 64·9% and its O.N. to 65, the addition of 3 c.c. lead bringing this to 75.

Illinois Pipe-line Crude yields 16% of 250° E.Pt. gasoline of 70 O.N. and 21·3% of naphtha or 37·3% of 400° E.Pt. gasoline of 54 O.N. and 9·7% kerosine. Cracking the reduced crude gave yields of 41·2–52·3% of gasoline of 68–69·5 O.N. and residues of No. 6 grade quality. Absorption and catalytic polymerization would yield 3·7% of gasoline, increasing the overall O.N. by 1·5.

Louden Crude is similar to Salem Crude, yielding 29·7% of 350° E.Pt. gasoline of 54·5 O.N., 7·0% of naphtha (or 36·7% of 420° E.Pt. gasoline of 49·0 O.N.), 8% kerosine, 10% gas-oil, 18% wax distillate, and 25·5% of bottoms of viscosity 468 S.U. at 210° F. Mount Carmel crude gives 36·2% of 400° E.Pt. gasoline of 55 O.N. and 10% of kerosine, and 53·7% of residue of cold test 40° F. and visc. S.U. at 100° F. of 457 secs. Sandoval crude yields 32·8% of 400° E.Pt. gasoline of 50 O.N. 11·0% kerosine, 26·9% gas-oil of cold test 45° F., and 29·2% of residue of cold test 60° F. and viscosity 199 secs. S.U. at 210° F. C. L. G.

36. Solutizer Process for Sweetening Gasolines. L. E. Border. *Oil Gas J.*, 18.7.40, **39** (10), 36.—Results obtained on a semi-commercial scale solutizer pilot plant, using the potassium salt of *iso*butyric acid dissolved in KOH for the extraction of mercaptans from various gasolines, are discussed. Extraction in a four-stage mixer-settler system and in a packed tower are compared, and two methods of regeneration of the solutizer solution studied. It was found that using the mixer-settler system the mercaptan sulphur (originally 0·05%) in 400° E.Pt. straight-run West Texas gasoline could not be reduced below 0·0017%, whereas in a packed tower a figure of 0·0004% was obtained. Extraction is improved by increasing solutizer concentration, and solutizer/gasoline ratio and reducing temperature, the extent of the effects being shown graphically. Loss of gasoline by solution in the spent solutizer is reduced in practice by diluting the latter with water, the gasoline released being returned to the main stream. The two methods of regeneration examined were: (1) direct injection of steam into the bottom of the stripping column, the overhead being condensed and both phases discarded, the stripped solution being pumped through the base of the tower, through the cooler when it is ready for use, and (2) a system of closed re-boiling in which the overhead aqueous condensate was returned to the top of the column as diluent and subsequently vaporized in the re-boiler to supply the stripping medium.

The first system has the following defects: (*a*) CO_2 from the steam accumulates in the solutizer, (*b*) mercaptans are present in the aqueous condensate and must be removed before discharge, and (*c*) the partial pressure of mercaptans is greater over a dilute caustic solution than over a concentrated solution, thus favouring the use of a dilute solution for the best stripping. The second system, using closed re-boiling, is free from these defects, since the stripping steam is free from CO_2, and the aqueous condensate is returned to the column while the solutizer is diluted before flowing down the column. No advantage was found in maintaining pressure in the regenerator. The adequacy of stripping of the solutizer can be tested by carrying out a doctor test on the solution shaken with mercaptan-free gasoline. Ten trays have been found enough when the closed system is used. Re-boiler tubes of 70·30 Cu·Ni alloy give adequate corrosion resistance, whilst stripped solution cooler tubes of stainless steel are perfectly smooth after 1 year's service, carbon-steel tubes corroding rapidly. The remainder of the system, apart from a slight metal loss in the vapour line, shows little evidence of corrosion.

Treating conditions for various gasolines were studied. The heavier cracked gasolines generally contain alkyl phenols, and are best treated with a "mixed" solutizer consisting of alkaline potassium *iso*butyrate containing alkyl phenols, whilst straight-run and light-cracked gasolines may be treated with the straight solutizer. Tabulated data on O.N. and lead susceptibilities of different types of solutized gasolines indicate that in practically every case the O.N. of the sweet unleaded gasoline was as high as or higher than that of the sour stock, whilst the lead susceptibility is unaltered. C. L. G.

Metering.

37.* Viscosity and its Effect on the Accuracy of Positive Displacement Metering. J. W. Donnell. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 13–21.—Viscosity is defined and viscous and turbulent flows are studied. Formulæ are derived for calculating

POLITECHNIKI

slippage losses for viscous flow in positive displacement metering, and examples are worked out in detail.

If the quantity of fluid forced through the meter is increased to a certain point, laminar flow through the slippage spaces will change to random fashion or turbulent flow. This point of transition has been found to vary with the following factors: size and shape of passage, density of fluid, velocity of fluid, and viscosity of fluid. These factors grouped together are known as the Reynolds number $Du\rho/\mu$, where D = Diameter in feet or a shape factor equal to four times the mean hydraulic radius; u = velocity in feet/sec.; ρ = density in lb./cu. ft.; and μ = viscosity expressed in lb./ft. sec. It has been found that streamlined flow can exist only when the Reynolds number is below 2100. As an example of this test for streamline flow, an illustration is worked out completely.

The paper is well illustrated. A. H. N.

38.* Fundamental Principles of Volume Meters. E. E. Ambrosius. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 22–26.—The principles of designing and operating fluid-meters are studied, and all types of meters are well illustrated by diagrams or photographs. The types of meters treated are classified under weighing, volumetric, and velocity meters. The first two classes are true "Quantity Meters," the distinctive feature of these meters being that all the fluid to be metered passes through the primary element in successive and more or less completely isolated quantities. The velocity meters belong to the rate-meter division through which the fluid flows in a continuous stream. The registration of these meters is derived from the action of the moving stream on the primary element.

The meters described are: (1) weighing or tilting-trap type; (2) volumetric, which include nutating piston, reciprocating piston (or simply piston), planetary piston, sliding or rotating vanes, gear or lobed impeller; (3) velocity meters, including helical and turbine meters. A. H. N.

39.* Some Fundamental Considerations in the Design and Application of Displacement Meters. E. W. Jacobson. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 29–33.—In the manufacture and application of displacement meters for measuring liquid hydrocarbons, extravagant claims of precise measurement have often been made. These have led to considerable trouble from failure of the meters to perform as claimed and from failure not only to check measurement by gauge-tanks, but also to check one meter against another. The limit of error most often quoted is 0·1%. This limit of error is examined as to its actual magnitude and the difficulty of obtaining measurement within this limit.

From the consideration of fundamental principles it is concluded that: (1) the claimed standard of measurement has been closer than commercially practical; (2) a close study of the characteristics of the two types of displacement meters—namely, pack and film sealed—indicates particular fields of application for each type, depending on accuracy of measurement required, permissible pressure drop, nature of register load, and range of viscosity of fluids to be metered; (3) the economics governing the installation of displacement meters must consider not only the cost of the meter, but also the additional cost of adequate liquid conditioning equipment, calibrating devices, and regularly scheduled maintenance, if satisfactory meter performance is to be obtained. A. H. N.

40.* Metering of Crude-Oil Production. E. O. Bennett. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 34–35.—Meters are precision-built pieces of machinery; and in order to perform with maximum reliability, they must receive proper attention from those familiar with their design and construction. With such attention, meters will precisely measure large volumes of oil with but little maintenance cost, and will save a large amount of money otherwise required for gauge-tanks and equipment.

Some pipe-line operators have meters installed on their systems with electric recording of all measurements in the dispatcher's office at headquarters. Such a system eliminates considerable personnel, and gives direct results at all times.

Meters are being more generally used in the producing industry as time goes on.

It is believed that their use will increase when operators understand and desire to take advantage of field stabilization or controlled evaporation losses of light high-gravity crudes made possible by the use of metering under pressure throughout a recovery plant. A. H. N.

41.* Calibration of Meters in Pipe-Line Service. H. Pohl and E. H. Counts. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 36–38.—Present knowledge of the use of positive displacement meters shows that they can be used for the measurement of crude oil and other petroleum products in pipe-line service if the counter mechanism is in proper adjustment, and the conditions under which the meter operates have been studied sufficiently, so the proper meter and auxiliary equipment needed for the particular job are used. Calibration of meters from time to time is necessary to correct errors in measurement due to wear, replacement of parts, changes in the physical properties of the oil, and in some cases to changes in operating conditions.

The calibration of meters can be accomplished with varying degrees of accuracy by comparing the volume registered by a meter against volumes shown by a calibrated proving tank, stock, or storage tanks, and against a master meter. Of the three methods, comparing meter readings against a calibrated proving tank is believed to be the most practical and accurate method for calibrating positive displacement meters in pipe-line service. A. H. N.

42.* Problems Encountered in Distillate Measurement. E. Kightlinger. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 39–41.—In most gas-fields it is impossible to locate the gasoline plant in such a manner that the liquid can be easily transported to the plant without its being carried through the gas-flow lines. Separators are installed at each well with the gas being drawn from the top of the separator and the distillate from a point near the bottom. The gas-flow is constant and measured through a standard orifice meter, while the liquid flow is governed by various type dump valves generally operated intermittently by a float within the separator. After the distillate passes through its meter it is carried through a separate line to a point downstream of the gas-meter, where the gas and liquid are again mixed in the flow-line. Both fluids are carried to the gasoline plant, where the distillate is again separated from the gas before the gas is taken into the plant for processing.

Due to load requirements, it is seldom possible to flow gas-wells at a steady volume for a long period, and at times it may be necessary to flow the wells at a low volume, due to prorated pulls, and thus help cause at least one, and maybe two, undesirable conditions.

The principal sources of trouble might be listed as follows : (1) Low gas differential across the orifice plate. (2) Fluid dump-valves not operating properly. (3) Paraffin. (4) Sand. (5) Water. (6) Hydrates.

These items are studied separately. It is concluded that gas-distillate measurement is still in its infancy, and for that reason many problems have not been solved to the entire satisfaction of the engineers of the manufacturing and operating companies. A. H. N.

43.* Displacement Meters in the Refinery. W. H. McCoy. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 42–48.—Displacement meters are used in the refineries to measure the rate of crude charge to stills, distillates from the stills to field tanks, fuel oil to burners, fuel oil from burners, liquid propane and butane, cooling oils to glands of hot-oil automatic-control valves, sealing oil to bearings and sealing glands of pumps, transfer of gasoline and other finished products from refinery to bulk terminals, transfer of finished products to distributing pipe-lines, barge calibration, crude sampling, steam condensate, water for cooling purposes, water for chlorination, kerosine, and other side-stream products being run to field, barrelling of oils, pilot-plant measurement, sulphuric acid in treating plants, ethyl fluid for treating gasoline and lubricating oil to bearings. The important items in this long list are treated separately.

In conclusion it is stated that at present the displacement meter is not extensively used in the refinery. This is chiefly because the units as now designed are not well adapted to refinery operation. Their cost of installation, the lack of flexibility, the inability to calibrate them in the field, the generally poor stuffing-boxes, the poor integrators, and the fact that the bodies, as a rule, do not conform to standard similar to those used by the refineries, all tend to make the refinery engineer hesitate to use this instrument. The poor metallurgical treatment of the working parts has also worked to the detriment of the unit. A. H. N.

44.* Progress in the Use of Meters in Crude Pipe-line Service. T. R. Aude. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 49–51.—Orifice meters, recording the rate of flow, have been found most useful when installed in the discharge lines from a pump station for checking the operation of the station and interpreting pressure drops. A decrease in pressure may be caused by a line failure or by trouble in the discharge pumps. A decline in the rate of flow as recorded by the orifice meter indicates that the pressure drop was caused by trouble in the pump station; an increase in the rate of flow with a drop in pressure indicates a line-break.

With regard to displacement meters, experience of 2 years demonstrates that displacement meters for crude-oil pipe-line service have progressed beyond the experimental stage. The cost of maintenance has been negligible. The wear on meters handling clean crude progresses slowly, and the resulting inaccuracies are within reasonable limits, and increase at a steady rate, making it a simple matter to apply a correction factor in figuring oil-runs. Their use has resulted in the elimination of the costs of tankage and labour for measuring oil. A. H. N.

45.* Development of Pipe-line Charts by Dimensional Analysis. L. H. Cherry. *Proc. Petrol. Fluid Metering Conf.*, 11.4.40, 58–64.—During the past twenty years numerous equations have been evolved to calculate variables involved in pipe-line transmission. A survey of recent articles shows the existence of two very apparent facts. First, that there has been a definite lack of correlation of the data recently published in these articles. Second, that, due to the many possible units for viscosity and density, many of the terms called Reynolds numbers, etc., are not the true terms. Equations are derived and curves drawn using such nomenclature, when actually the term used has a number of constant mixed into the true term, and is therefore not truly dimensionless.

The paper shows possible combinations of variables that may be derived by means of dimensional analysis, and submits charts combining these variable combinations in their correct functions according to the latest data available. The basis of the charts is the Fanning equation: $p = 2f\frac{L}{D}\rho\frac{V^2}{G}$.

Weymouth's formula is criticized and an example solved by Weymouth's formula and by means of the charts shows a difference of 17·8% between the two solutions. It is implied that Weymouth's formula is in error. A. H. N.

Chemistry and Physics of Petroleum.

46. *X*-Ray Examination of Poly*iso*butylene. C. S. Fuller, C. J. Frosch and N. R. Pape. *J. Amer. chem. Soc.*, 1940, **62**, 1905–1913.—Brill and Halle (*Naturwissenschafen*, 1938, **26**, 12) found that when poly*iso*butylene of high molecular weight was stretched to the maximum, the amorphous *X*-ray diagram was replaced by a fibre pattern. The work of the authors confirms this finding. Evidence is presented suggesting 1 : 3 arrangement of the methyl-group pairs along the chain and a helical chain configuration.

Natural rubber and poly*iso*butylene show an analogous effect of elongation on their *X*-ray patterns. The fibre pattern of rubber is very weak at elongation of about 100%, and becomes progressively more intense as more crystallinity is produced by further stretching. Poly*iso*butylene of medium degree of polymerization does not show crystallinity on stretching except at elongation above 1000%.

The *X*-ray structure of poly*iso*butylene crystals is discussed, and the paper is illustrated with photographs and diagrams. T. C. G. T.

47. Nitrogen Compounds in Petroleum Distillate. XVIII. Isolation, Ozonization, and Synthesis of 2 : 4-Dimethyl-8-*s*.-butylquinoline. L. M. Schenck and J. R. Bailey. *J. Amer chem. Soc..*, 1940, **62**, 1967–1969.—Continuing their work on the isolation of kero bases from 320–330° kero base fraction, the authors have succeeded in obtaining yields of a new $C_{15}H_{19}N$ kero base, 2 : 4-dimethyl-8-*s*.-butylquinoline. The structure was determined and the compound synthesized from acetylacetone and *o*-amino-*s*.-butylbenzene. T. C. G. T.

48. Fluorinated Derivatives of Ethane and Ethylene. VI. Corrective Data. A. L. Henne and E. G. Wiest, *J. Amer. chem. Soc.*, 1940, **62**, 2051–2052.—Improved physical data are listed for $CCl_2 = CF_2$,CCl_3CClF_2, CCl_2BrCF_2Br, CClFBrCClFBr, and CCl_3CF_3. T. C. G. T.

49. Catalytic Dehydrogenation of Representative Hydrocarbons. A. G. Oblad, R. F. Marschner, and L. Heard. *J. Amer. chem. Soc.*, 1940, **62**, 2066–2069.—Hydrocarbons of all classes have been dehydrogenated over two types of chromic oxide catalyst and at the conditions : temperature 480° C. ; space velocity 0·5 ; duration, 3·0 hr.

*cyclo*Hexanes are converted into aromatics, so, too, are paraffins, but much less rapidly than the *cyclo*hexanes. In the case of paraffins the activity of the catalyst steadily decreases and elementary carbon is deposited. *cyclo*Pentanes dehydrogenate slowly, and resemble olefines in depositing large amounts of carbon.

Simple methylated aromatic hydrocarbons are unaffected, but alkyl aromatics with longer side-chains, such as ethylbenzene, are slowly dehydrogenated to products which strongly poison the catalyst. The residue responsible for the poisoning is a tar rather than carbon. T. C. G. T.

50. Infra-Red Absorption Studies of Some Hydrocarbons. F. T. Wall and G. W. McMillan, *J. Amer, chem. Soc.*, 1940, **62**, 2225–2227.—The infra-red absorption spectra of a number of hydrocarbons in carbon tetrachloride solution have been studied in the 3·35 μ region. The positions of the absorption maxima have been correlated with the aromatic and aliphatic properties of the molecules. T. C. G. T.

51. Effect of Surface on Cool Flames in the Oxidation of Propane. R. A. Day and R. N. Pease. *J. Amer. chem. Soc.*, 1940, **62**, 2234–2237.—There have been several attempts to explain the occurrence of successive " cool flames " during the oxidation of paraffin hydrocarbons. Higher aldehydes, the surface of the reaction vessel, and peroxides have all been advanced as explanatory.

The work of Harris and Egerton indicates that the peroxides are not responsible, and in earlier work (*J. Amer. chem. Soc.*, 1934, **56**, 2034) one of the authors had reached a similar conclusion when he found that by coating a Pyrex reaction vessel with potassium chloride the yield of peroxides could be largely suppressed without materially altering the rate of oxidation of the propane.

In this work the authors find that the cool-flame region in 1 : 1 propane–oxygen mixtures is not materially affected by etching the reaction tube or coating it with potassium chloride. It is therefore determined by conditions in the gas phase. Attention is directed to the region of negative temperature coefficient of the rate as an effective control, leading to successive flames and preventing ignition.

T. C. G. T.

52. Quenching of Fluorescence in Solution. II. Temperature and Solvent Effects. R. W. Stoughton and G. K. Rollefson. *J. Amer. chem. Soc.*, 1940, **62**, 2264–2268.—The effects of changes of temperature, viscosity, and dielectric constant of the solvent on the quenching of the fluorescence in solution have been studied. The fluorescent substances used were quinine sulphate, fluorescein, and acridone, whilst the quenchers were potassium chloride and iodide. Water glycol, glycerol, and methyl alcohol, but not oil, were used as solvent. T. C. G. T.

53. Hydrogen Bonds Involving the C–H Link. X. The Solubility of Donor Solutes in Halogenated Hydrocarbons. C. S. Marvel, F. C. Dietz, and M. J. Copley. *J. Amer. chem. Soc.*, 1940, **62**, 2273–2275.—Organic compounds containing donor centres generally are much more soluble in hydrogen-containing halogenated hydrocarbons than in completely halogenated hydrocarbons. These higher solubilities are attributed to solute–solvent association by means of $CH \leftarrow O$ or $CH \leftarrow N$ bonds. T. C. G. T.

54. Synthesis of 4 : 5-Dimethylchrysene. M. S. Newman. *J. Amer. chem. Soc.*, 1940, **62**, 2295–2300.—In a study of the relationship between chemical constitution and carcinogenic activity of hydrocarbons, 4 : 5-dimethylchrysene has been synthesized. Evidence supporting the structure is presented. T. C. G. T.

55. Spreading of Binary Mixtures of Volatile Organic Liquids on Water. C. P. Keim and E. R. Washburn. *J. Amer. chem. Soc.*, 1940, **62**, 2318–2320.—Work on the measurement by means of a surface-film balance of the spreading pressures of pure organic liquids on water (cf. I.P. Abs. 1940, No. 1497) has been extended to cover certain binary mixtures such as benzene–toluene, benzene–*cyclo*hexane, benzene–nitrobenzene, and benzene–bromobenzene. T. C. G. T.

56. Notes on the Preparation and Properties of some Aliphatic Hydrocarbons. L. Schmerling, B. S. Freedman, and V. N. Ipatieff. *J. Amer. chem. Soc.*, 1940, **62**, 2446–2447.—The synthesis of three hexanes, one heptane, and three octanes is described. Their boiling points, refractive indices, densities, and octane numbers are given.

2-Methylpentane (C.F.R. Octane No. 71·5) was prepared via 2-methyl-pentanol-4, which was obtained by hydrogenating mesityl oxide.

2 : 2-Dimethylbutane (Octane No. 93) was synthesized by the hydrogenation of *tert.*-butylethylene, which was obtained by the pyrolysis of pinacolyl acetate.

2 : 3-Dimethylbutane (Octane No. 94) was prepared by two methods. The first consisted of hydrogenating dimethylbutadiene obtained by the dehydration of pinacol over alumina at 427° C. The second method consisted of the reduction of pinacolone to pinacolyl alcohol, the dehydration of the alcohol by means of oxalic acid to a mixture of tetramethylethylene and *unsym.*-methyl*iso*propylethylene, and the hydrogenation of these olefins to the desired hexane.

The carbinols which were intermediate in the preparation of 2 : 2 : 3-trimethylbutane (Octane No. 100), 3 : 4-dimethylhexane (Octane No. 84·5), 2 : 2 : 3-trimethylpentane (Octane No. 100), and 2 : 3 : 4-trimethylpentane (Octane No. 94·5), were synthesized by means of the Grignard reaction. The carbinols were then dehydrated and hydrogenated. T. C. G. T.

57. Nature of the Surface of Catalytic Nickel. W. W. Russell and W. V. Loebenstein. *J. Amer. chem. Soc.*, 1940, **62**, 2573–2580.—An account is presented of the selective poisoning of nickel catalysts, and the information obtained on the nature of the active surface indicating it to be non-uniform is discussed. T. C. G. T.

58. Polyalkylbenzenes. XXVII. Preparation of Pure Ethylbenzenes. L. I. Smith and C. O. Guss. *J. Amer. chem. Soc.*, 1940, **62**, 2625–2629.—It has been found that the ethylation of benzene by ethyl chloride and aluminium chloride occurs quite as readily as does the corresponding methylation, and that it is possible to control the alkylation so that the major portion of the product consists of any desired polyethylbenzene. Tri-, tetra-, penta-, and hexa-ethylbenzenes have been prepared by the method.

As in the case of methylation, no vicinal isomers resulted from these ethylations.
T. C. G. T.

59. Polyalkylbenzenes. XXVIII. Physical Properties of the Tetraethylbenzenes. L. I. Smith and C. O. Guss. *J. Amer. chem. Soc.*, 1940, **62**, 2630–2631.—Three tetraethylbenzenes have been highly purified and their physical properties determined: *sym.*-tetraethylbenzene, b.pt. 246·8° C. (734 mm.); f.pt. + 10° C.; n_D^{20} 1·5054, and d^{20} 0·8788; *unsym.*-tetraethylbenzene, b.pt. 247·4° C. (734 mm.); f.pt. – 21° C.; n_D^{20} 1·5056; and d^{20} 0·8799.

Vicinal tetraethylbenzene, b.pt. 251·1° C. (734 mm.); f.pt., fluid at – 50° C.; n_D^{20} 1·5125; and d^{20} 0·8875.

Vapour pressures are also presented. T. C. G. T.

60. Jacobsen Reaction. VII. Polyethylbenzenes. L. I. Smith and C. O. Guss. *J. Amer. chem. Soc.*, 1940, **62**, 2631–2635.—To obtain information concerning the

Jacobsen rearrangement of ethylated benzenes, six polyethylbenzenes, 1 : 2 : 4- and 1 : 3 : 5-triethylbenzenes, the three tetraethylbenzenes and pentaethylbenzene have been studied. T. C. G. T.

61. Polyalkylbenzenes. XXX. Nitration of Tetra-, Penta-, and Hexa-ethylbenzenes ; Bromination of the Tetraethylbenzenes. L. I. Smith and C. O. Guss. *J. Amer. chem. Soc.*, 1940, **62**, 2635 ;2638.—Nitration of polyalkylbenzenes is of interest not only because the reaction provides good derivatives for identification of these hydrocarbons, but also because of the curious attack on the side-chain, leading to the formation of nitrates, which often accompanies it. Bromination, on the other hand, while a smooth reaction, does not lead to good derivatives in the polymethylbenzene series. Nitration and bromination of three tetraethylbenzenes and penta- and hexa-ethylbenzenes have been studied and the reactions and products compared with those of the corresponding polymethylbenzenes. T. C. G. T.

62. Polyalkylbenzenes. XXXI. Preparation and Physical Properties of 1 : 2 : 3-Trimethylbenzene (Hemimellithene). L. I Smith and L. J. Spillane. *J. Amer. chem. Soc.*, 1940, **62**, 2639–2642.—1 : 2 : 3-Trimethylbenzene (hemimellithene) is not formed when xylenes are methylated by methyl chloride and aluminium chloride, nor can it be obtained from the other trimethylbenzenes by a Jacobsen rearrangement. Several methods have been used for this synthesis, but the authors have used a modification of the Tawney–Agre method which, utilizing the Tiffeneau rearrangement, prepared the compound from benzyl chloride.

The physical properties of the purified product were : d^{20}_4 0·8944 (d^0_4, d^{25}_4, and d^{30}_4 are also presented), n^{20}_D 1·5130 (n_D is also reported at 5, 10, 15, 25, and 30° C.) and f.pt. – 25·41° C. Vapour-pressure/temperature relationships are also presented. T. C. G. T.

63. Cyclization of Dienynes. VIII. Ring Closures with α- **and** β-*cyclo***hexenyl-acetylene Derivatives of Octalin.** C. S. Marvel, D. E. Pearson, and L. A. Patterson. *J. Amer. chem. Soc.*, 1940, **62**, 2659–2665.—The cyclization reaction which occurs when substituted dienynes are treated with sulphuric acid has now been extended to some α and β derivatives of octahydronaphthalene. T. C. G. T.

64. Structure of Vinyl Polymers. IX. Catalysts. C. S. Marvel and E. H. Riddle. *J. Amer. chem. Soc.*, 1940, **62**, 2666–2670.—Evidence is presented showing that vinyl polymers formed by the action of catalysts such as peroxides and boron fluoride or by photochemical activation have the same arrangement of monomeric units in the polymer chain. T. C. G. T.

65. Sulfhydryl and Cysteine Derivatives of 1 : 2-Benzanthracene, 10-methyl-1 : 2-benzanthracene and 3 : 4-Benzpyrene. J. L. Wood and L. F. Fieser. *J. Amer. chem. Soc.*, 1940, **62**, 2674–2681.—The sulfhydryl and cysteine derivatives of highly carcinogenic hydrocarbons such as 1 : 2-benzanthracene, 10-methyl-1 : 2-benzanthracene and 3 : 4-benzpyrene offer a source of information concerning the nature and significance of the reaction involved in the metabolism of carcinogenic hydrocarbons.

The dihydroxydibenzanthracene synthesized by the authors is believed to be identical with the substance excreted by carcinogenic-hydrocarbon-injected mice and rats. As the excreted substance is the product of natural detoxification, any measures which promote that conversion should be advantageous. T. C. G. T.

66. Macropolymerization : the Mechanism of Activation. E. P. Irany. *J. Amer. chem. Soc.*, 1940, **62**, 2690–2698.—Macropolymerization is usually explained as a chain reaction which is supposed to be initiated and propagated by radicals produced by the opening of the double bond of the monomer. This conception is inadequate for many reasons, which are discussed. The evidence for the discussion is based largely on styrene polymerization. T. C. G. T.

67.* Studies on the Separation of Paraffin Waxes. Part 1. Principles Involved in the Fractional Melting of Wax. M. F. Sawyer, T. G. Hunter, and A. W. Nash. *J. Inst. Petrol.*, 1940, **26** (202), 390–400.—In this, the first part of the paper, a fundamental basis of classifying and identifying fractions of wax in equilibrium experiments is discussed. A " calculated molecular weight " of wax is explained, together with its use in the experiments which are to be reported in later parts. A. H. N.

68.* Chlorination of Methane for the Preparation of Carbon Tetrachloride. A. I. Kiprianov and T. S. Kusner. *Oil Gas J.*, 18.1.40, **38** (36), 49.—Investigations have been carried out on an improvement of the method of Jones, Allison, and Meighan (*Ind. Eng. Chem.*, **11**, 639 (1919)) for the production of CCl_4 by direct chlorination of methane at 400° C. in the presence of activated carbon as catalyst. This method avoids the danger of ignition of the reacting gases when excess Cl_2 is used, but the maximum velocity of Cl_2 attained is too slow for commercial exploitation (12 litres per 100 gm. of activated carbon). Increasing the velocity caused ignition, but it was found that by diluting the reacting gases with inert gases this could be avoided. In practice natural gas containing CO_2 and N_2 could be used. The reaction takes place in a narrow zone, so that widening the combustion tube is an advantage. The highest volume velocity (160 litres Cl_2 per 100 gm. of carbon per hour) was attained using 32 litres CH_4, 128 litres Cl_2, and 250 litres HCl, giving a yield of 94% of the theoretical, with only slight incandescence in the tube. Tests showed that there was no danger of the gas mixture exploding during chlorination. C. L. G.

69.* Temperature-Gravity Graph for Gravity Determinations. O. G. Vanderveer. *Petrol. Engr.*, Dec. 1939, **11** (3), 56.—A graph is reproduced enabling the gravity of petroleum oils between 10 and 100 to be converted from that at any temperature between 0° and 180° F. to that at any other temperature. It is based on the A.P.I. gravity tables in the Tag Manual, the smooth curves being drawn between the points on the graph. Differences resulting from this are, however, small for the variations in temperature normally encountered. For accurate gravity determinations, as for purchasing, direct determinations of the gravity of the oil brought to 60° F. is advisable. C. L. G.

70.* The Dependence of Viscosity of Liquids on Constitution. A. H. Nissan, L. V. W. Clark, and A. W. Nash. *J. Inst. Petrol.*, 1940, **26** (198), 155–211.—A study of the correlation of viscosity and molecular shape of liquids is presented. Liquids appear to obey one law of viscosity–temperature variation, independently of whether they are associated, unassociated, or dissociated. Molecular shape appears to be of greater importance than molecular weight in determining the " specific viscosity curve." A. H. N.

Analysis and Testing.

71.* Burning Test for Kerosine. I.P.T. Serial Designation—K 35(T). Anon. *J. Inst. Petrol.*, 1940, **26** (196), 91–95.—This method supersedes that issued in 1938. A. H. N.

72.* Simple Petroleum Tables for Use with Density Hydrometers. V. Stott. *J. Inst. Petrol.*, 1940, **26** (199), 235–245.—Tables showing change of density of petroleum oils with temperature and for conversion of density in g./ml. to weight in air in lb./Imp. gal. are given. A note is appended on the accuracy of density determinations by means of British Standard density hydrometers. A. H. N.

73.* Analysis and Testing of Lubricating Greases. M. W. Webber. *J. Inst. Petrol.*, 1940, **26** (200), 273–293.—The paper deals solely with soap greases, and discusses the determination of soap base and content, mineral-oil content, free acid and alkali, water content, unsaponified fat, ash, impurities, fillers and addition agents, corrosion, melting point, consistency, stability at high and normal temperatures and at high pressures and on storage, load-carrying capacity, and low-temperature efficiency. A. H. N.

See also Abstract No. 85.

Motor Fuels.

74.* Alternative Fuel. L. van Lottom, *Auto. Engr,* 1940, **30** (399), 219–220.—For motor vehicles, butane has the advantage of a relatively low vapour pressure, and thus does not require a high-pressure storage cylinder. However, the cooling effect of evaporation leads to starting difficulties in cold weather, whilst the higher temperatures under the bonnet may cause vapour lock. These difficulties may be overcome by the use of *iso*butane or a 35/65 mixture of propane and butane.

It was found that 28 lb. of butane was equivalent in heat value to 3·89 lb. of petrol. Tests on an E.6 Ricardo engine showed that the highest compression ratios at which petrol and butane could be used were 5·5 : 1 and 8·5 : 1, respectively. Maximum power was 11·9 h.p. for butane and 10·5 h.p. for petrol. Consumption of butane, expressed as equivalent pints of petrol per B.H.P. hour, was somewhat higher than with petrol. K. A.

75.* Octane Ratings of a Number of Pure Hydrocarbons and of some of their Binary Mixtures. J. Smittenberg, H. Hoog, B. H. Moerbeek, and M. J. v.d. Zijden. *J. Inst. Petrol.,* 1940, **26** (200), 294–303.—C.F.R.–A.S.T.M.-Motor method octane numbers have been determined of fifty hydrocarbons, the majority of which were in a very pure state, and of certain binary mixtures. A method is described by means of which the octane numbers below zero and above 100 were estimated. A. H. N.

Lubricants and Lubrication.

76. Study of Monolayers of some Esters and Chlorinated Derivatives Possibly Useful as Lubricating Addition Agents. G. L. Clark and J. V. Robinson. *J. Amer. chem. Soc.,* 1940, **62,** 1948–1951.—The work is inspired by the oil industry's need for knowledge of the mechanism of action of certain polar organic compounds, which, when added to lubricating oil, increase its " oilness." Of the various theories of this mechanism the one considered in this investigation is that the addition agent is absorbed on the metal, acting as a buffer between metal surfaces. *X*-Ray diffraction has been used previously by one of the authors to show that such an absorption actually takes place.

The monomolecular film balance was selected as a tool to furnish quantitative data on the strength of absorption, and the nature of the molecular packing of material used as " oilness " agents.

It is suggested that the collapse-pressure of monomolecular films measured on the hydrophil balance may be correlated with the resistance to shear of the same substances oriented on a metal surface in an engine bearing. It is also suggested that a surface consisting of a close-packed array of hydrogen atoms, attached to hydrocarbon chains, should offer the minimum hindrance to the hydrocarbon molecules of a lubricating oil slipping over it.

With an improved film balance under carefully controlled conditions, measurements are made of area per molecule and collapse pressure on water surfaces for monolayers of certain esters which have possible use as addition agents in lubricating oils—namely, methyl stearate, L-chlorostearate, dichlorostearate, oleate ricinoleate and chlororicinoleate, ethylene glycol distearate and ricinoleate and tricresyl phosphate.
T. C. G. T.

77.* Notes on Filtration and Distribution of Lubricating Oil. T. C. Worth. *J. Inst. Aut. Engrs,* Nov. 1940, **9** (2), 13.—Contaminants which affect the properties of used oils are considered, and figures for analyses of used oils from a petrol and a diesel engine are given. Methods of oil conditioning include gravitation, straining, adsorption, filter aids, and electrostatic precipitation, with filtering divided into suction, strainer, full flow, by-pass, and batch-filtration methods, each being considered in that order. With the exception of adsorption and filtration, other than the batch-method, the methods are impracticable in an automobile engine. That adsorption is the chief factor in cleaning oil by filtration is the conclusion reached from experiments on the effectiveness of various materials for use in filters. The possibility of chemical aids to produce a really clean oil is discussed, as is the location of the various types of filter, and it is stated that, in the majority of cases, a full-flow filter is no more than a badly designed by-pass filter.

A method of testing filters and examining pressure drop is described, as well as the difficulties of obtaining a representative material for tests. In an appendix an electrical phenomenon is described which appears to have a direct relation to filtration and the material used for the filter, and which shows a maximum value at a definite pressure. This pressure coincides with that used for many by-pass filters.

H. L. W.

78.* Static Friction of Lubricated Surfaces. A. Fogg and S. A. Hunwicks. *J. Inst. Petrol.*, 1940, **26** (195), 1–18.—Details and conclusions of an investigation carried out to obtain information on boundary friction and the boundary lubricating properties, or oiliness, of various substances are presented. Oiliness appears to be unconnected with viscosity of the fluid; but it is connected with the properties of the boundary layer. A. H. N.

79.* Catalytic Effect of Metals on the Ageing of Lubricating Oil for Diesel Engines. H. L. Matthijsen. *J. Inst. Petrol.*, 1940, **26** (196), 72–90.—Catalytic effect of all relevant metals with which lubricating oil comes into contact in the Maybach diesel engine is examined and the results are reported. All the ferrous metals are found to stimulate ageing, whilst the effects of non-ferrous metals are either nil or negative.

A. H. N.

80.* Some Experiments on Seizure between Lubricated Hard Steel Balls. D. Clayton. *J. Inst. Petrol.*, 1940, **26** (199), 256–271.—Experiments and results are described, to find the effect of change of the test conditions on the behaviour of lubricants in the four-ball apparatus. Similarly, the effects of mechanical adjustments in the machine itself on seizure and lubrication results are reported. A. H. N.

Asphalt and Bitumen.

81. Revised Specifications for Cut-back Asphalts. Anon. Asphalt Institute Construction Series No. 51, 1940.—The Asphalt Institute specifications for cut-back asphalts have been revised in the light of experience gained during the past seven years. The three types of material, R.C., H.C., and S.C., have been divided into six grades, and viscosities adjusted, so that each product of the same grade is of the same viscosity, the upper limit is twice the lower, and a gap exists between consecutive grades. For R.C. and M.C. materials the amount of distillate received at specified temperatures is expressed as a percentage of the total recovered at 680° F., a minimum recovery at 500° F. is now included, the minimum recovery at 600° F. has been increased for R.C. materials, and the maximum recovery at 437° F. and 500° F. for M.C. materials is lower than minimum figures for R.C. products.

A minimum is prescribed for percentage of residue from distillation, the lower penetration limits have been increased to 80 for R.C. materials and to 120 for M.C. materials, and the minimum ductility has been increased to 100. In the case of residues of more than 200 penetration the material is acceptable if the ductility is over 100 at 60° F. The limits in the solubility test are retained, but carbon tetrachloride is to be used instead of carbon disulphide. The specifications are tabulated and indications given as to the product to be used for particular purposes and of the working temperatures. A. O.

82. Revised Specifications for Slow-curing Liquid Asphaltic Road Materials. Anon. Asphalt Institute Construction Series No. 52, 1940.—The revised Asphalt Institute specifications for slow-curing materials are tabulated, and it is pointed out that these include requirements for percentage and ductility of 100 penetration asphalt residue, maximum and minimum limits for the flood test on residue from distillation, and freedom from water in all but the two highest grades. A. O.

83. Specifications for Emulsified Asphalts. Anon. Asphalt Institute Construction Series No. 53, 1940.—Specifications for rapid, medium, and slow-setting emulsions are tabulated, and an indication is given as to the principal uses of the emulsions. The methods of testing are those described in the A.S.T.M. methods of Testing. A. O.

84.* Stability of Emulsions, Part III. General Survey of Solid Emulsifying Agents, with Special Reference to the Hydrous Oxides and Hydroxides. H. L. Bennister, A. King, and R. K. Thimas. *J. Soc. Chem. Ind.*, 1940, **59**, 226–232.—It is pointed out that to give oil-in-water emulsions the solid agent used must be more easily wetted by water than by oil, and vice versa for water-in-oil emulsions. Among materials which have been used for the former are plaster of Paris, silica, lime, calcium carbonate, fine clays, and powdered coal, and for the latter, carbon-black. The efficiency of emulsification depends on the particle size of the emulsifier, which also determines the size of the emulsified droplets. Previous work is discussed, and it is pointed out that solid emulsifiers have only a limited general application, the most common being bentonite for asphaltic bitumen emulsions. The solid emulsifying agents in general give very coarse unstable emulsions. Unlike Parts I and II, the present work is only a qualitative survey. A gelatinous precipitate was found to be usually much more efficient than a granular preparation of the same material. The authors examined many inorganic solids, and concluded that the hydrous oxides and hydroxides were the most effective. The oxides and hydroxides of seventeen metals and silicon were precipitated by a number of methods and their emulsifying properties examined, and the results are given in detail, suitable symbols being used to grade the resultant 50% emulsions of carbon tetrachloride, paraffin, and carbon disulphide. The emulsifying efficiency varied very much with the method of precipitation, very gelatinous, highly dispersed systems being in general most effective. Many of the hydroxide precipitates improved or deteriorated on ageing, and aged aluminium hydroxide was found to be particularly good. Emulsions stabilized by gelatinous hydroxides were found to be relatively coarse, although very stable and insensitive to the presence of electrolytes, and with a pronounced tendency to cream and form " clumps." As a general rule, it appeared that for a given compound those specimens precipitated at either very high or very low concentration had maximum emulsifying capacity, and ammonia appeared to be the best precipitating agent. Froth formation was noticeably absent with all these emulsions. Magnesium and aluminium compounds were especially efficient, whilst copper hydroxide should be useful owing to its additional value as a fungicide. Addition of traces of alkali, minute amounts of soaps, and other surface-active compounds considerably improves the dispersions, as does the precipitation of the hydroxides from solutions containing about 1% of hydrophilic colloids. The hydrous precipitates also possessed the ability to wet substances, such as sulphur and various pigments, which are not easily wetted. Gelatinous precipitates other than oxides or hydroxides were found to be ineffective—*e.g.*, silica gel, magnesium, zinc, and aluminium silicates. It was concluded that the basic character of the oxides and hydroxides was a fundamental property necessary for emulsification. The paper concludes with the experimental details of the methods of preparing some of the hydroxides. H. G. W.

85. Flow Properties of Asphaltic Bitumens with Reference to Road Behaviour. A. R. Lee and J. B. Warren. *Proc. Ass. Asph. Pav. Technol.*, 1940, **2**, 340–364.—The effect of stress and temperature on the viscosity of different types of bitumen has been examined as part of an investigation of the significant physical properties of bitumen in relation to road behaviour. A similar examination of the flow properties of asphaltic road mixtures is in progress, and a study is being made of the behaviour of asphaltic materials under normal traffic conditions.

The instruments used to cover the range of viscosities between atmospheric and mixing temperatures were: a rotating coni-cylindrical viscometer, an Ostwald viscometer for opaque viscous liquids, and a falling cylinder viscometer. These instruments are discussed. Measurements on a 65 pen. Mexican bitumen showed that viscosity diminished with increasing shearing stress at temperatures below 70° C., whilst at higher temperatures the bitumen behaved as a Newtonian liquid.

The relation between shearing stress and rate of shear did not obey the Bingham law $D = 1/\eta(S - S_0)$, and could not be represented by other equations, but could be expressed to a first approximation by $D = kS^{\rho}$, k and ρ being constants, except for bitumens deviating markedly from Newtonian flow.

It is thought that an equation introducing one term representing Newtonian flow and a second term representing additional flow due to breakdown of the structure would be more logical, but without a value for viscosity at zero stress this cannot be satisfactorily checked.

The value of ρ in $D = kS^{\rho}$ gives a measure of the deviation from Newtonian flow and provides a method of expressing the degree of plasticity.

Examination of a number of 65 pen. bitumens indicated a tendency for asphaltene and plastic flow index to increase together.

Differences in viscosity of bitumens of the same penetration are said to affect directly the mechanical properties of surfacing materials containing the bitumens.

In discussing the effect of temperature on viscosity it is pointed out that bituminous materials present particular difficulty in attempts to obtain a value for the temperature susceptibility of viscosity, owing to their relatively large temperature susceptibilities. It has been found that the equation $k = AT^{-n}$, where T is temperature °F. and A and n are constants, holds for the range from about 20° C. to 90° C. The coefficient n, the slope of the straight line obtained on plotting log k against log T, is termed the logarithmic temperature coefficient, and provides a criterion for distinguishing between the relative susceptibilities of bitumens. No relation was found between asphaltene content and the temperature coefficient.

Specimens of road-surfacing materials have been found to exhibit similar log temperature coefficients, when tested under conditions of steady stress, to those of the bitumens, and it is concluded that the susceptibility of the road mix is determined by that of the binder. It is stated that it is not clear what significance the elastic properties of a bituminous binder have in respect of road behaviour. The elastic properties are most prominent when the stress is rapidly applied, but even under conditions of continuously-applied constant stress elastic recovery occurs on the sudden removal of the stress. The amount of recovery depends on the stress applied and the degree to which the material has been previously sheared.

The evaluation of the elastic constants is said to offer some difficulty, but the total recovery gives an adequate means of distinguishing this property. Results obtained indicate that the elastic properties are directly related to the asphaltene content. A. O.

86. Developments in Research and Technology of Bituminous Road Materials. G. W. Eckert. *Proc. Ass. Asph. Pav. Technol.*, 1940, **2**, 365–431.—The author reviews the literature dealing with the manufacture, properties, testing, and application of bituminous materials published during 1939. A. O.

87.* Physical Constants of Asphaltic Bitumens. Part 1. R. N. J. Saal, W. Heukelom, and P. C. Blokker. *J. Inst. Petrol.*, 1940, **26** (195), 29–39.—A survey is given of the following physical properties of asphaltic bitumens : specific gravity, coefficient of cubical expansion, specific heat, thermal conductivity, permeability to water vapour, surface tension, and total surface energy.

The methods by which these properties have been determined are described in detail. A. H. N.

88.* Flow Properties of Bituminous Materials. A. R. Lee, J. B. Warren, and D. B. Waters. *J. Inst. Petrol.*, 1940, **26** (197), 101–128.—The effects of stress and temperature on the flow properties of various types of bitumens and bitumen-aggregate mixtures are reported, as obtained by a viscometer and by beam and tensile tests. A. H. N.

89.* Physical Constants of Asphaltic Bitumens. Part 2. J. Saal, K. Meinema, and P. C. Blokker. *J. Inst. Petrol.*, 1940, **26** (199), 246–255.—A survey of the following electrical properties of asphaltic bitumens is made : dielectric strength, angle of dielectric loss, dielectric constant, and electrical conductivity. The methods by which these properties have been determined are described. A. H. N.

90.* Use of a Bitumen Emulsion in Soil Stabilization. A. E. Lawrence. *J. Inst. Petrol.*, 1940, **26** (201), 357–364.—The underlying principles of soil stabilization are discussed, and certain conclusions, based on American investigation and practice, are quoted. A. H. N.

91.* Soil Mechanics in Foundation Engineering. R. L. James. *J. Inst. Petrol.*, 1940, **26** (201), 365–374.—The subject is analysed from structural engineering point of view and certain conclusions are detailed. A. H. N.

92.* Manufacture and Testing of Roofing-Felts and Damp-courses. D. M. Wilson. *J. Inst. Petrol.*, 1940, **26** (202), 377–389.—Testing methods to evaluate the qualities of roofing-felts and damp-courses are described and the manufacture of these materials is described and well illustrated by photographs. A. H. N.

93.* Researches on Asphaltenes. Part 1. F. J. Nellensteyn and J. P. Kuipers. *J. Inst. Petrol.*, 1940, **26** (202), 401–406.—The solubility of asphaltic bitumen in solvents having the same surface tension (ether and petroleum spirit) is made the subject of detailed study. It appears ether should be used in preference to normal gasoline for asphaltene determination. A. H. N.

Special Products.

94.* Hazards Associated with Butadiene and its Peroxides. D. A. Scott. *J. Inst. Petrol.*, 1940, **26** (199), 272.—Butadiene may form explosive compounds on heating under pressure, or on contact with air or oxygen giving explosive peroxides. Storage over caustic soda or washing with the alkali frees butadiene from its peroxides and their hazards. A. H. N.

95. Isolation and Identification of Fluoranthene from Carbon Black. J. Rehner. *J. Amer. chem. Soc.*, 1940, **62**, 2243–2244.—It is known that fluoranthene can be prepared by the dry distillation of coal or coal-tar, or by the passage of acetylene and hydrogen through a hot porcelain tube. It has also been demonstrated that the waxy material deposited from the hydrogen stream during the cracking of natural gas consists partly of acenaphthylene and pyrene.

The author has isolated fluoranthene in the hydrocarbon-soluble fraction of carbon produced by the thermatomic process. T. C. G. T.

See also Abstract No. 93.

Detonation and Engines.

96.* The Manufacture and Use of Tetraethyl-Lead. G. Edgar. *J. Inst. Petrol.*, **26** (197), 146–149.—The chemistry of the manufacture as well as historical and operational reviews of the use of tetraethyl-lead are presented. A. H. N.

97. Fuel Economy in Petrol Engines. W. T. David and A. S. Leah. *J. Instn. Mech. Engrs*, Oct. 1940, and *Proc.*, 1940, **143** (5), 289–312.—Petrol engines differ widely in their performance, even when due allowance is made for differences in compression ratio, mixture strength, and the nature of the fuel. The object of the paper is to provide data whereby the margin of improvement practically possible for any given engine may be estimated, and to suggest broadly how it may be secured.

The paper is divided into three parts. In Part I are given charts from which may be found the indicated thermal efficiency, fuel consumption per I.H.P.-hour and I.M.E.P. which are attainable in a compact combustion-chambered engine of any bore between 3 in. and 8 in., for compression ratios between 4 : 1 and 9 : 1, speeds from 1000 r.p.m. upwards, and mixture strengths between 20% weak and 20% rich. Separate charts are presented for octane, ethyl alcohol, and benzene. Hence data in regard to any mixed petrol may be deduced with fair accuracy.

The charts are based on the accurate gaseous internal energies, entropies, and dissociation constants derived from spectroscopic data by means of quantum statistics. Allowance has been made for unavoidable heat losses during explosion and expansion, using heat-loss estimates deduced from closed-vessel explosion experiments and single-cylinder engine-test results. The comparison of the performance of actual engines with the attainable efficiencies shown by the charts yields a measure of the margin of improvement practically possible.

Comparison of the charts shows that, for the same suction temperature, the attainable fuel consumption (lbs. per I.H.P.-hour) is some 10% less for octane than for benzene, although the latter can attain 2½% greater power. With ethyl alcohol the attainable power is very slightly less than with octane, whilst the attainable fuel consumption is very much higher.

Included in Part I are tabulated data on internal energies, constant-volume entropy values, and dissociation constants for gases entering into the combustion reactions. In Part II various phenomena associated with the working fluid and the combustion of the charge are discussed. These include turbulence and its effect on heat loss during explosion and expansion, the delay period after ignition, incomplete combustion at the peak pressure, and after-burning. It is deduced that, in an engine of 4·5-in. bore running at 1500 r.p.m., the heat loss would be about 1% (of the heat of combustion of the fuel burnt) during explosion and about 5% during expansion. Under detonating conditions the rate of heat loss is greatly increased; this is mainly attributable to greatly augmented turbulence, caused by the sudden expansion of the detonated gas into the gas previously burnt.

Part III is a discussion of actual engine performance compared with attainable performance. An example is quoted of a "good" engine, yielding an indicated thermal efficiency of 35·7%. The charts show an attainable efficiency of 38·0%. The defect in efficiency is thus 2·3% of the heat of combustion. This could be accounted for on the assumption that only 70% of the heat energy of combustion had been released at the moment of attaining maximum pressure. If perfect mixing of the charge could be secured, an increase in power of at least 6·25% would be realized.

Apart from distribution difficulties in multi-cylinder engines, it is believed that improvement in charge mixing within each cylinder would be one of the major lines along which progress could be made. K. A.

98. Problem of Valve-stem and Valve-head Deposits. A. T. Colwell. *J. Soc. aut. Engrs*, Sept. 1940, **47** (3), 358.—Whilst much attention has been directed to varnish and lacquer deposits, little thought appears to have been given to valve deposits. The latter may contain lead in various forms, as well as silica, iron oxide, insolubles, and dirt impurities. The real cause of the trouble is the fuel and partly burned lubricating oil, which, collecting on the upper part of the stem, eventually holds the valve open. A valve may continue to operate satisfactorily over a long period when seating properly, but once it is unseated, trouble always occurs. The remedies which have been applied suggest that each engine has individual characteristics, more or less oil having beneficial effects in different cases. The types of deposit are enumerated, and it is shown that oils appear to have a critical temperature at which they leave a maximum deposit. This fact suggests that by controlling the temperature of the valve and metering the oil supply, the deposits should be kept below harmful proportions.

Remedies found useful in the aircraft field are described; metering the oil and eliminating water and acid vapours have proved beneficial. Sodium-cooled valves have reduced one type of deposit, but, unless the guide is thoroughly cooled, may cause varnish on the stems. The low-temperature driving with automotive engines, as well as the variety of fuels and oils used, make the problem more difficult in this case. The oil has not always been found to be the cause of the trouble, but in some cases the fuel is directly responsible, particularly in lean-mixture operation, and many mechanical alterations have been found effective, as also have solvents. Cool running—*e.g.*, many stops and starts with short runs in between—provide the condition for maximum build-up. The author concludes that insufficient attention has been paid to this problem by those workers concerned with fuels and lubricants.

H. L. W.

99. Control of Smoke in the Automotive Diesel. W. W. Manville, G. H. Cloud, A. J. Blackwood, and W. J. Sweeney. *J. Soc. aut. Engrs*, 1940, **47** (4), 397–406.—The acceptance of automotive diesel engines in the U.S.A., particularly for bus service, has been retarded by smoky exhausts and users have tended to resort to lighter fuels, which have been found to effect an improvement. Since English engines give satisfactory results on the heavier fuels, a study of the possible factors controlling smoke was undertaken.

Three laboratory engines of representative type were used, and smoke density was measured by a photo-electric device. The influence of different factors on smoke was investigated both under equilibrium and accelerating conditions. Field tests also were run on a variety of equipment.

Some fifty fuels were tested, and it was established that the most important fuel characteristic influencing smoke is volatility. Whereas the lighter fuels cause less smoke, they also give less power. The conclusion was reached that the major part of the smoke reduction generally attributed to change in fuel is actually due to the resultant change in power output. At equivalent power outputs there are only small differences in the smoking tendencies of fuels covering a wide range of fuel volatility. The effect of cetane number is less important than that of volatility, except in cases where the ignition quality is below the minimum demanded by considerations of roughness and cold starting.

Whilst the original purpose of the study was to concentrate on the relation between fuel properties and smoking, it became apparent that operating conditions, engine design, mechanical condition, and adjustment might far outweigh any possible effect of fuel.

It is concluded that freedom from smoke can be achieved only by accepting a limitation on power output, whether this is brought about by resetting the fuel-pump adjustment or by the use of lighter fuels. It would appear more desirable to reset the smoke stop on the pump whilst retaining the economic advantage of the heavier fuel, which will have the higher calorific value per gallon. This conclusion is substantiated by the London buses, which develop no objectionable smoke on fuels of 550° F. mid-boiling point, but which have power units of sufficient size to permit working below the critical power point. K. A.

Coal and Shale.

100.* Classification of Oil Shales and Cannel Coals. A. L. Down and G. W. Himus. *J. Inst. Petrol.*, 1940, **26** (201), 329–348.—Attention is directed to the confusion which exists in the nomenclature and classification of naturally occurring oil-yielding materials. A system of nomenclature based on the principal plant and mineral constituents, and a classification showing the inter-relationships and gradation of properties of these substances, are put forward. A. H. N.

BOOK REVIEWS.

The Design of High-Pressure Plant and the Properties of Fluids at High Pressures. By Dudley M. Newitt. Pp. viii + 491. Oxford University Press. 1940. Price 35*s*.

During the past twenty years the successful application of high pressures to the chemical industry has resulted in a very considerable study of the properties of matter and of the kinetics of chemical reactions at high pressures. In this book Professor Newitt has confined himself to describing the effects of pressure on physical processes occurring in fluid systems.

The book is divided into two parts. Part I is devoted to the design of high-pressure equipment, and commences with a chapter on materials of construction, followed by one on gas cylinders. Chapter III, on "The Calculation of Stresses and Strains in Pressure Vessels," leads up to a very informative discussion on "The Design of High-Pressure Plant and Equipment" in Chapter IV. Joints, valves, compressors, pumps and safety devices are dealt with, together with the use of glass in high-pressure work. Part I concludes with a chapter on "The Measurement of High Pressures." The material dealt with and the examples of vessels and auxiliary parts presented are such as would be employed in laboratory or small-scale experimental work. This section of the book is obviously written as a result of academic rather than industrial experience, and certain minor problems in technique which quite frequently occur in industrial high-pressure work are not included. The heating of high-pressure vessels, the design of glands and of flow-meters for measuring fluid flows, for example, are not dealt with. While such matters are in the main minor problems, they can be and have been in the past major problems in high-pressure work. One has only to recall the difficulties encountered by Bergius in the heating of his semi-scale hydrogenation convertors, and in the design of a satisfactory gland for these same reaction vessels, to appreciate the important part played by such matters in high-pressure work. Apart from these, the measurement of fluid flows, particularly small gas flows, at high pressures, would make a useful and attractive addition to the book.

The thirteen chapters of Part II deal almost exclusively with the physical properties of fluids at high pressures. After a short introduction dealing with the kinetic theory, the P.V.T. relationships of gases and the equation of state problem are discussed. Recent compressibility work and the volumetric behaviour of the more important gases, some of their binary mixtures, and one ternary mixture are described. The liquefaction of gases, and the use of enthalpy–entropy and temperature–entropy diagrams in the design of gas compressors and circulators are discussed.

The influence of pressure on the viscosity, the dielectric strength and refractivity of gases is reviewed in two chapters, whilst another describes the effect of pressure on the viscosity and refractivity of liquids. The final chapter describes the P.V.T. relationships of liquids.

Tables of data on the thermodynamic properties of hydrogen, nitrogen, carbon monoxide, carbon dioxide, and methane constitute Appendix I. Appendix II is mainly concerned with data on the Joule–Thompson coefficient for nitrogen, air, helium, and argon, whilst Appendix III tabulates the refractive index of carbon dioxide. Numerous data in tabular and diagram form are also included in the text.

It is very obvious from a perusal of Part II that, as pointed out in the Preface, "there is still a serious lack of fundamental data of all kinds relating to condensed systems." The very clear and concise way in which Professor Newitt has presented the existing knowledge on the fundamental physical properties of fluids under high pressure in this volume should, however, do much to encourage further exploration of this fascinating and important field of research, whilst the description of experimental equipment and methods included will be found particularly helpful to investigators engaged on such high-pressure research. T. G. HUNTER.

Symposium on New Materials in Transportation. American Society for Testing Materials, Philadelphia, Pa. Pp. 94. Price $1.50.

On a number of occasions recently the American Society for Testing Materials has organized a series of papers bringing together a number of otherwise unconnected industries from some common standpoint. This is particularly appropriate to the A.S.T.M., by reason of the wide range of subjects which come within its sphere, and the papers presented are by the foremost chemists and engineers in the various industries, and are always of special interest.

"Symposium on New Materials in Transportation" represents a series of papers presented at the Detroit Spring Meeting in March 1940, and includes the use of steel and rubber in the automotive industry, of concrete and asphalt in transportation, of fuels and lubricants, together with the first article in this series, which is less general than the other—namely, "Exhaust-Valve Materials for Internal-Combustion Engines." The treatment adopted in the different articles is not the same, but whilst some review general progress and research developments, others are primarily concerned with the developments which are taking place in specifications. Thus in the paper by D. P. Barnard and A. H. Fox, on "Trends in the Properties of Volatile Liquid Fuels," these are discussed from the point of view of specifications and methods of test developed by the A.S.T.M., and the whole review is almost entirely from the American point of view. A series of interesting charts show the trends in properties for motor fuel, domestic furnace oils, and furnace oils over a long period of years. Developments in the automobile industry are given in comparison with the dates when the A.S.T.M. adopted various types of test for motor fuels, and a series of supplementary charts show changes in the properties of various gasolines over the period 1905–1940. These show the continual increase in the anti-knock characteristics of regular gasolines, particularly over the period 1930–1940.

While this is a general review, there are a number of statements made, particularly as regards aviation gasoline, which are not in agreement with the standard methods of test adopted in the U.S. specifications, and from this point of view the article will be found particularly interesting.

"Developments in Lubrication," by J. P. Stewart, R. C. Moran, and O. M. Reiff, is primarily concerned with one specific aspect—namely, the development of certain types of additive for use in automobile oils. On the other hand, the article by B. E. Gray, of the Asphalt Institute, on "Recent Developments in the Use of Asphalt for Transportation Purposes," is a general review of the subject, and will be found to be of particular interest. The subject is treated from a wide viewpoint, dealing with such factors as subgrade design, influence of traffic on design, width of pavement aggregate, together with trends in plant mixtures and special considerations applying to airports and railroads. In this article, again, an independent viewpoint has been taken, and for this reason the treatment is specially welcome, since it leads to a re-examination of ideas currently accepted. Although only three articles have been reviewed in detail, the other reviews are of considerable value to petroleum technologists, since the problems affecting other industries are often of great importance to those primarily interested in petroleum. F. H. GARNER.

BIBLIOTEKA POLITECHNIKI ŚLĄSKIEJ

INSTITUTE NOTES.

JANUARY 1941.

FORTHCOMING MEETINGS.

Wednesday, February 5th, at 2.15 p.m. "Addition Agents for Lubricating Oils," by E. A. Evans, M.I.A.E., F.C.S. (Chairman, Lubricating Oil Group).

This meeting will be held at the Institution of Mechanical Engineers, Storey's Gate, London, S.W.1.

CONSISTENCY OF GREASE.

The Grease Panel of the Institute (Chairman Mr. E. A. Evans) has for some time been considering the whole question of the standardization of methods of testing greases. Members and companies are invited to send to the Assistant Secretary a description of their own procedure for the determination of the *consistency of grease.* It is hoped to have available for consideration by the Panel a complete survey of the methods employed for this determination. Any comments on the methods or results of experience will be of considerable value to the Panel in their discussions and will be welcomed.

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.

CROOK, Roy Dallas, B.Sc., Chemist, Shell Company of Australia, Adelaide, S. Australia. (*T. Hartigan ; H. A. Banks.*)

FORD, Alexander Langridge, B.Sc., Chemist, Anglo-American Oil Co., Ltd. (*C. Chilvers ; E. B. Evans.*)

MACKIE, William Frederick Charles, Shell Marketing & Refining Co. (*G. Davidson ; E. LeQ. Herbert.*) (*Trans. to Fellow.*)

Oosten, William Hennanus (Dutch), General Manager, Bataafsche Petroleum Mij., Middlesex. (*J. Kewley ; J. H. Blakiston.*)

Penard, Herbert F. L. (Dutch), Consulting Engineer, 53 Clifford's Inn, London, W.C.2. (*G. H. Coxon ; C. E. Spearing.*)

Withers, George, Lubrication Engineer, Texas Co. (S.A.), Ltd., Johannesburg. (*W. E. Gooday ; E. W. Ramsay.*)

Applications for admission as Students.

Driver, Antony Victor, Shell Marketing & Refining Co's Laboratory. (*H. E. Priston ; R. G. Strickland.*)

Keen, Sidney, Student, Royal School of Mines. (*S. E. Coomber ; G. D. Hobson.*)

Poole, Walter George, Student, Royal School of Mines. (*S. E. Coomber ; G. D. Hobson.*)

Sasson, Albert, Student, Royal School of Mines. (*S. E. Coomber ; G. D. Hobson.*)

ARTHUR W. EASTLAKE,
Honorary Secretary.

LIST OF ADVERTISERS.

	PAGE
Audley Engineering Co., Ltd.	—
C. R. Averill, Ltd.	Inside back cover
Babcock & Wilcox, Ltd.	vi
Baker Oil Tools Inc.	xiii
Cardwell Mfg. Co.	iv
W. Christie & Grey, Ltd.	iii
A. F. Craig & Co., Ltd.	xi
Foster Wheeler, Ltd.	—
W. J. Fraser & Co., Ltd.	ix
Hadfields, Ltd.	iii
Institute of Petroleum	v
International Paint & Compositions Co., Ltd.	v
Lucey Export Corporation	xiv
Lummus Co.	xii
National Savings Committee	x
National Supply Corporation	vii
Nordberg Manufacturing Co.	—
Oil Well Supply Co.	—
John G. Stein & Co., Ltd.	viii
Stewarts and Lloyds, Ltd.	Back cover
Tintometer, Ltd.	—
Worthington Simpson, Ltd.	—

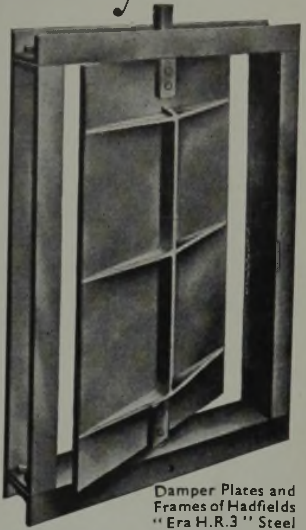
Steels specially suitable for the Oil Industry

ERA TRADE MARK **MANGANESE STEEL** for Sprocket Wheels, Pulleys, etc

ERA C.R. **NON-CORRODING STEEL** for Thermowells

ERA H.R. **HEAT-RESISTING STEELS** for Tube Supports, Heat Exchangers, etc.

HADFIELDS LTD.
EAST HECLA AND HECLA WORKS
SHEFFIELD · · · · · ENGLAND



Damper Plates and Frames of Hadfields "Era H.R.3" Steel

No. 2334

CONTINUOUS WASHING

Holley Mott Plants are efficiently and continuously washing millions of gallons of Petroleum products daily. Designed for any capacity. May we submit schemes to suit your needs ?

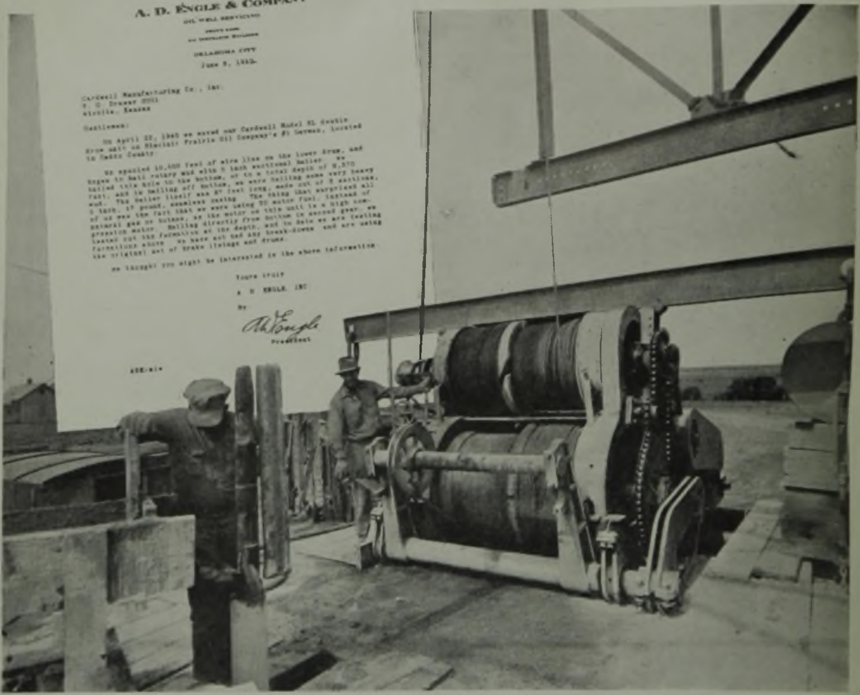
HOLLEY HM MOTT

Continuous Counter-Current Plant

World-Wide Licensees, W. CHRISTIE & GREY LTD.
FOUR LLOYDS AVENUE, LONDON, E.C.3.

Your DEEP WELL *Servicing Problems*

CAN BE SOLVED . .


A. D. ENGLE & COMPANY

OKLAHOMA CITY

with a "Cardwell" Rig . .

FOUR WAYS A "CARDWELL" HELPS SOLVE YOUR PROBLEMS

Transmission—The new "Cardwell" five-speed, even-step transmission of 1450 foot-pound torque capacity (240 h.p.) has a single lever gear shift and requires only one movement to shift from low to high. This results in faster operation when running or pulling tubing or drill pipe.

Friction Clutches are mounted on the countershaft to transmit the power directly to the drums and are shielded against oil and water. They may be engaged quickly or slowly as the job demands, saving time at each operation and absorbing the shock load of each engagement. More than 500 of these friction clutches have been placed in use since 1936 without a single part failure. There is a jaw clutch in each drum for providing full floating drums.

Double Safety Brakes—The "Cardwell" brakes are water-cooled and designed so either brake will automatically operate independently and hold the full capacity load, yet they are fully equalized and have a single adjustment. The brake flanges are rolled alloy steel, heat-treated and ground, with three to four times the life of ordinary steel flanges. The smooth, polished hard surface gives more than twice the ordinary brake lining life.

Low Cost Haulage and Rig-Up Time—The lightest and strongest of alloys are used in the all-steel construction of this rig. The engine and hoist are mounted on one skid to facilitate handling. Every wearing part of this "Cardwell" rig is heat-treated to the last pin and pin-hole in the control mechanism. Light weight results in low hauling costs.

CARDWELL ALL STEEL
THIS TRADE MARK INSURES HIGHEST QUALITY AT LOWEST PRICE

CARDWELL MFG. CO. INC.
FORMERLY ALLSTEEL PRODUCTS MFG. CO.
P. O. [illegible] · Cable Address: "ALLSTEEL", WICHITA · "CARDSTEEL", NEW YORK
Wichita, Kansas, U. S. A.

● We build 16 models of servicing winches and drilling rigs — Send your well depths and details of your well equipment for recommended model and size. We have a standard model for any drilling problem or operating method.

F. J. OLSEN, Export Manager, 570 Lexington Aven., N.Y. City. Cable Address: "Cardsteel."

PAINTS FOR THE PETROLEUM INDUSTRY

REGD TRADE MARK

"DANBOLINE SILVERETTE" *The super aluminium paint for all refinery purposes. Only actual experience can prove its amazing durability.*

"TANCTECTOL" *The only protective paint for the INTERIOR of petroleum storage tanks. Withstands permanent immersion in all petroleum fractions, benzole and salt or fresh water. Used by the British Admiralty, Royal Air Force, and leading Oil Companies.*

Write for free booklet "PAINT IN THE OIL INDUSTRY."

INTERNATIONAL PAINT & COMPOSITIONS Co., Ltd.

U.S. Enquiries:
21 WEST ST.,
NEW YORK.

GROSVENOR GARDENS HOUSE
GROSVENOR GARDENS
LONDON, S.W.1

Main Factory
FELLING-ON-TYNE
ENGLAND

ANNUAL REVIEWS OF PETROLEUM TECHNOLOGY

Vol. 5 (Covering 1939)

Authoritative reviews of developments during 1939 and early 1940 in the petroleum and allied industries. Each chapter is accompanied by an extensive bibliography.

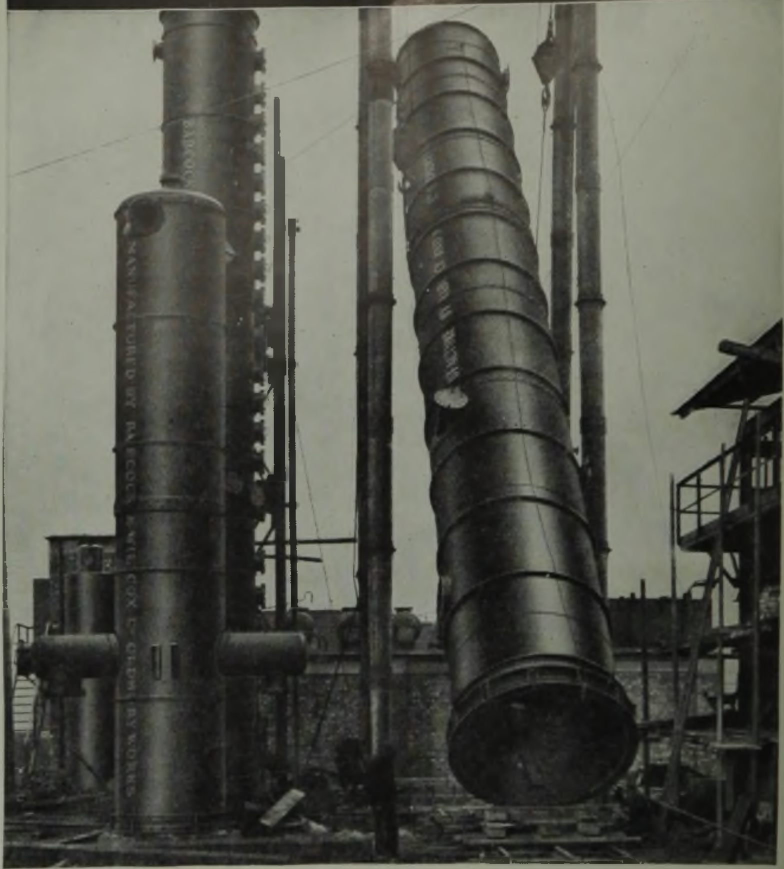
Cloth Bound pp. viii + 444

Price 11s. *post free*

Obtainable from

THE INSTITUTE OF PETROLEUM
c/o THE UNIVERSITY OF BIRMINGHAM
EDGBASTON, BIRMINGHAM 15

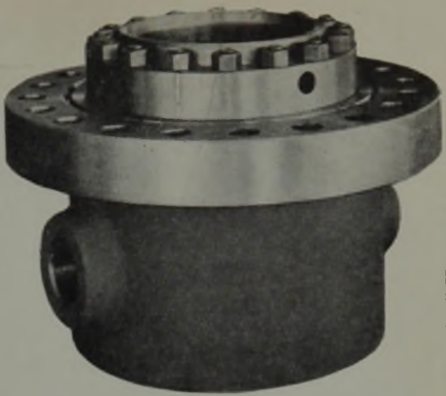
SPECIAL VESSELS OF ALL KINDS



OUR erection facilities throughout the world are unsurpassed. The illustration shows a Vacuum Fractionating Column being lifted into position. Our Works are particularly well equipped for the construction of re-action chambers, coking chambers, flash chambers, fractionating columns, strippers, evaporators, heat exchangers, condensers and all vessels included in oil refinery and treatment plants, digesters for pulp mills, etc. All Babcock Welded Vessels are manufactured to **LLOYDS, API-ASME, ASME,** or other recognised codes and are subject to inspection by all the leading insurance companies or other official bodies.

BABCOCK & WILCOX LIMITED
BABCOCK HOUSE, 34 FARRINGDON ST., LONDON, E.C.4

Type JH-1 Casing Heads to support and pack off long strings of Casings against high pressure.



Type EH-3 has a flange at the upper end permitting the attachment of blow out preventers or flanged tubing or casing heads.

NATIONAL
CASING
HEADS

Types JH and KH Casing Heads are of the slip suspension type permitting setting casings at any depth without cutting special nipples. Packing is located above slips and is independent of the weight of the casing.

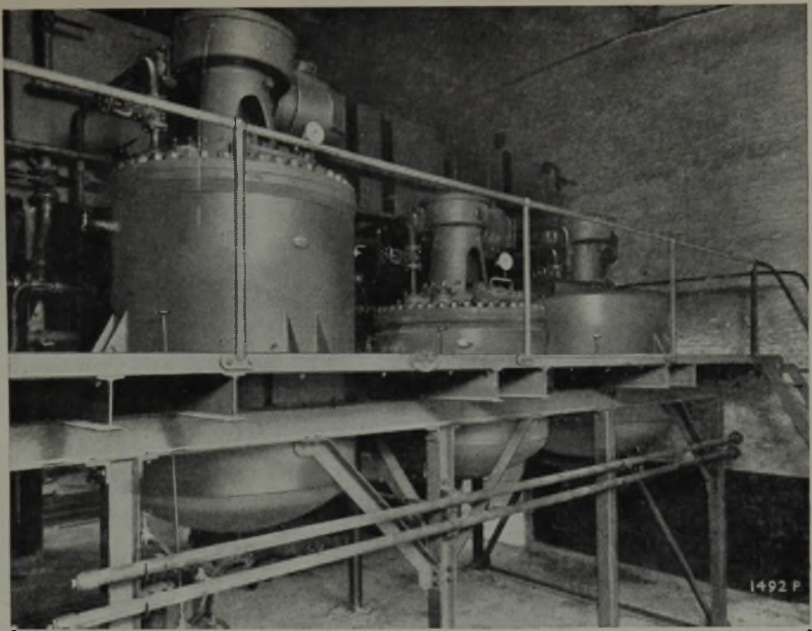
Type EH Casing Head is of the mandrel suspension type. In the patented packing arrangement a single gland compresses two rows of narrow asbestos packing.

Type KH-1 is for medium pressure service.

NATIONAL
OWECO
RIVER PLATE HOUSE LONDON E.C.2.

THE varying needs of heat-using industries are fully met by our wide range of high quality refractories. For severe conditions in oil firing we recommend the use of NETTLE firebrick (42/44% alumina) and STEIN SILLIMANITE (63% alumina).

JOHN G. STEIN & Cº Lᵀᴰ *Bonnybridge Scotland*



Fraser Pressure Saponification Plant for Grease

HIGH TEMPERATURE HEATING

Many processes call for high temperature heating, the commonly used methods such as steam heating, hot oil circulation and direct firing, each being associated with certain disadvantages. Within the temperature range 200°C to 400°C these are largely overcome by using the

DOWTHERM SYSTEM

of Vapour-Phase Heating. DOWTHERM Vapour is generated in a boiler and is used like steam, giving closely controlled high temperatures at low pressures. The modern tendency for high temperature saponification opens a field for Dowtherm heating. The Dowtherm boiler and all auxiliary equipment are designed and fabricated by W. J. Fraser & Co. Ltd.

By FRASERS of DAGENHAM

W. J. FRASER & CO. LTD., DAGENHAM, ESSEX

TAS/FZ. 236

Financial Patriotism

CHEAP BORROWING BY THE STATE *lightens the Nation's financial burden and so gives more power to defeat the enemy and to maintain our industry and commerce both during and after the war. The Government needs* NOW *vast sums of money loaned in the form of 2½% National War Bonds. The urgency of the need — the greatness of the cause — make inexorable demands on us all.*

2½% NATIONAL WAR BONDS

(1945-47). A full Trustee Security — Price of issue £100 per cent — Subscriptions of £100 or multiples of £100 will be received until further notice — Interest accrues from date of purchase — Prospectus and application forms obtainable from Banks or Stockbrokers.

Issued by The National Savings Committee, London

This page has been placed at the disposal of the National Savings Committee by the Council of the Institute

LACHMAN VAPOUR PHASE TREATING PROCESS

* * *

LACHMAN TREATED spirit does not require any inhibitor.

LACHMAN TREATING in a single operation conserves anti-knock quality; reduces gum content to the vanishing point; reduces sludge and polymerization losses to the minimum and reduces sulphur.

The practical advantages also of a method which is fool-proof in the sense that it cannot be overdone must appeal to all refiners.

* * *

A. F. CRAIG & CO., LTD.

PAISLEY

Representing:

VAPOUR TREATING PROCESSES INC.,
555, South Flower Street,
Los Angeles CALIFORNIA

THE WINKLER-KOCH ENGINEERING CO.,
335, West Lewis Street,
Wichita KANSAS

Another...

LUMMUS

CRACKING UNIT

Accepted

This one after 18 days of initial run

For a major oil company Lummus recently completed a Lummus Combination Three-Coil Cracking Unit . . . viscosity-breaking, gas oil and heavy naphtha reforming. » » » Eighteen days after the unit was put on stream it was completely accepted, having met all guarantees. The initial run was continued to 25 days, when the unit was shut down for inspection purposes. » » » This recently completed unit — the ninth consecutive Lummus Cracking Unit to be accepted during initial firing runs of 25 days or more — is equipped with Lummus Floor-Fired, Raised Hearth Heaters, with improved steam generation feature in the convection section. Provision is also made for steam generation from waste heat.

W. H. JONES

Representing: **THE LUMMUS COMPANY**

83 Barn Hill, Wembley Park, Middlesex

BAKER "BAKWIK" DRILL PIPE FLOATS

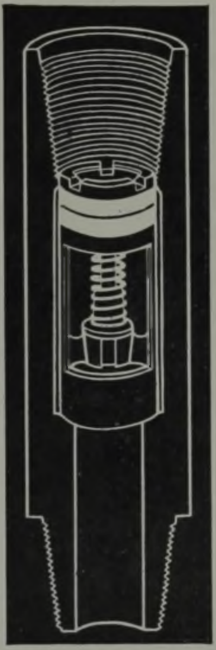
Pay Their Own Way by:

1. **Minimizing Plugged Bits**
2. **Relieving Strain on Derrick, Rig Equipment and Wire Lines**
3. **Eliminating Wet Strings**
4. **Providing Positive Back-Pressure Valve**
5. **Preventing Blowouts**
6. **Minimizing — Often Preventing Entirely — Irreparable Damage Should Drilling String Part**

SIMPLEST, MOST EFFICIENT TYPE OF VALVE ASSEMBLY

Valve Assemblies for "Bakwik" Drill Pipe Floats are designed for *Simplicity, Ease of Handling* and *Positive Operation. There are no threads* . . . Entire Valve Assembly is dropped into the Float Joint Body *by hand* . . . and removed in the same manner. No wrenches or special tools are required.

IT WILL PAY YOU to Secure Complete Details From the Nearest Baker Office or Representative. See Pages 266-268 of the 1940 Composite Catalog.

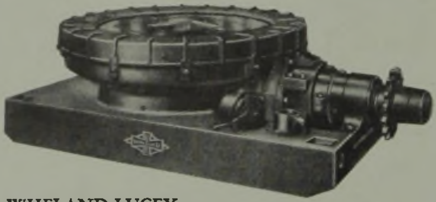


BAKER OIL TOOLS, INC.

MAIN OFFICE AND FACTORY: 6000 South Boyle Avenue
Box 127, Vernon Station, Los Angeles, California
CENTRAL DIVISION OFFICE and FACTORY: 6023 Navgiation Blvd.
Box 3048, Houston, Texas
EXPORT SALES OFFICE: 19 Rector Street, New York, N. Y.

ROTARIES YOU CAN RELY ON

- Bailing Lines
- Boiler Insulating Casings
- Cable Tool Drilling Lines
- Casing Lines
- Complete Drilling Rig Installations
- Crown Blocks
- Diesel Mechanical Hook-ups
- Diesel Electrical-Mechanical Hook-ups
- Drawworks
- Drilling Engines
- Drilling and Fishing Tools
- Gas Burners
- Lapwelded Steel Casing
- Oil Bath Swivels
- Oil Well Boilers
- Oil Well Chains
- Oil Well Tubing



WHELAND-LUCEY
TOTALLY ENCLOSED 17½″ OR 27½″
TRIPLE OIL BATH ROTARIES

Specially designed for drilling the deepest wells, these Rotaries can be relied upon to perform better, and to be safer and longer lived at the highest speeds because they are triple lubricated. Three separate oiling compartments permit the use of the proper type of lubricant for the bearing contained in that compartment. Thus, main bearings, thrust bearings, roller bearings and gear and pinion all operate with a minimum of friction and consequently last longer.

All parts of these heavy duty Rotaries are of specially selected annealed steel.

For simplicity, strength and service specify these Rotaries.

Send for Bulletin No. 149 for full description.

- Pipe Reconditioning Equipment
- Power Slush Pumps
- Pull Rods
- Reverse Clutches
- Rotaries
- Rotary Drilling Lines
- Rotary Drill Pipe
- Seamless Steel Casing
- Shale Separators
- Steam Slush Pumps
- Steel Derricks
- Steel Tanks
- Sucker Rods
- Trailers
- Traveling Blocks
- Turbo Generators
- Wire Rope
- Wrought Iron Pipe

LUCEY EXPORT CORPORATION

Incorporated in Delaware, U. S. A., with Limited Liability.

OIL WELL SUPPLIES

BROAD STREET HOUSE, E. C. 2 LONDON, ENGLAND

3505 Woolworth Building, New York, N.Y.
811 Sterling Building, Houston, Texas

Calle Defensa 320, Buenos Aires, Argentina
58 High St., San Fernando, Trinidad, B.W.I.