

## VISCOSITY MEASUREMENT AND VISCOSITY INDEX.

By J. C. CRAGG, B.Sc., A.I.C., and E. A. EVANS (Member of Council).

THE assessment of the viscosity temperature characteristics of petroleum products in terms of viscosity index is gaining in popularity. Its recent appearance in Government specifications alone makes its estimation a matter of considerable importance to the petroleum industry. It becomes necessary, therefore, to see clearly how accurately viscosity can be determined, and what effect errors have on the calculated viscosity index. The recent publication of the fourth edition of "Standard Methods for Testing Petroleum and its Products" contains a recommended method for the measurement of viscosity in absolute units, and a considered statement that the apparatus should measure viscosity with an error not exceeding 0.3 per cent. The adoption of this method, with its implied reproducibility, introduces such a change, that a review may be of assistance to those whose acquaintance with it may not be close. To impose a standard of accuracy is laudable, if it can be attained with reasonable skill and equipment. The petroleum industry in America has had experience with a similar method,<sup>2</sup> and believes that it can reasonably maintain an even greater precision than that imposed by the Institute of Petroleum. It will be interesting to observe the agreement forthcoming between workers in this country and the U.S.A. when they measure the viscosity and viscosity index of identical petroleum oils.

Viscosity index<sup>3</sup> is an empirical number and is calculated from the equation :

$$\text{V.I.} = \frac{L - U}{L - H} \times 100$$

where

$U$  = viscosity at 100° F. of the oil under examination ;

$L$  = viscosity at 100° F. of an oil of V.I. = 0  
(selected Texas Coastal type) ;

$H$  = viscosity at 100° F. of an oil of V.I. = 100  
(selected Penna type) ;

all three oils having the same viscosity at 210° F. The calculation of viscosity index was originally based upon Saybolt units.<sup>4</sup> Consequently if the accuracy obtainable with the Saybolt viscometer is insufficient, and this instrument is abandoned and replaced by another instrument of greater precision, viscosity index must be based either on other units or on conversions to Saybolt units. The system based on kinematic units<sup>5</sup> is now generally accepted as being superior to Saybolt seconds.

Before proceeding, it is convenient to know the numerical deviations in viscosity index created by errors in viscosity determinations. Table I shows the possible deviation from the mean in V.I. units for a specified viscosity error.





TABLE I.

Viscosity at 210° F., centistokes.	Possible deviation from mean in V.I. units for stated viscosity error.							
	0.25%.		0.5%.		1%.		2%.	
	V.I. 0.	V.I. 100.	V.I. 0.	V.I. 100.	V.I. 0.	V.I. 100.	V.I. 0.	V.I. 100.
2.0	3	3	6	5	12	9	24	18
4.0	3	2	6	4	12	7	23	13
6.0	2	1	4	3	8	5	16	9
8.0	2	1	4	2	7	3	13	7
12.0	2	1	3	1	6	3	11	5
20.0	1	1	2	1	5	2	10	4

As the theme of the paper is founded upon this conception, its meaning should be made clear by means of a diagram (Fig. 1).

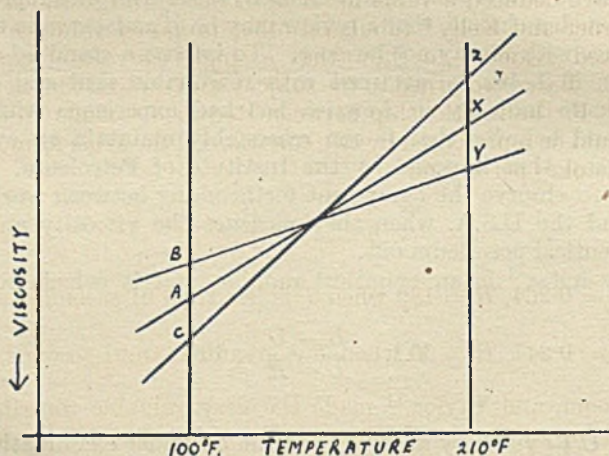


FIG. 1.

*AB AC, XY, and XZ* represent stated viscosity error ( $x$  per cent.).

*AX* represents the viscosity characteristics giving the true or mean V.I.

*BY* represents the viscosity characteristics giving the maximum V.I. obtainable with an error of  $x$  per cent.

*CZ* represents the viscosity characteristics giving the minimum V.I. obtainable with an error of  $x$  per cent.

In this country Redwood viscosity<sup>6</sup> is accepted in most trading transactions. The weaknesses of Redwood viscometers have been discovered, and much time and thought have been expended upon improvement. Finally it was agreed to accept Redwood viscometer No. 1307 as the standard. The reproducibility obtainable with N.P.L. certified No. 1 Redwood viscometers is stated by the I.P.<sup>6</sup> to be 1 per cent. of the mean value for efflux times of over 100 seconds at a temperature not exceeding 140° F., while at a temperature of 200° F., and also for viscosities lower than 100



seconds, the viscosity should not differ from the mean value by more than one second. This precision is sufficiently good for works control, has been satisfactory for trading, and is within the requirements demanded by efficient lubrication. Moreover the instrument is convenient to manipulate. Since recognized tables do not exist for the calculation of V.I. from Redwood seconds, it is necessary to convert to kinematic units, in order to calculate V.I.

The conversion of Redwood seconds to kinematic units was first investigated by Higgins,<sup>7</sup> who, in 1913 put forward the formula :

$$v = AT^m - B/T$$

where

$$A = 0.00260$$

$$B = 1.715$$

and

$$T = \text{Redwood time.}$$

Later Barr<sup>8</sup> pointed out that if Higgins had ignored one abnormal result he would have deduced a value for  $A$  of 0.00255, and so sailed nearer the truth. Garner and Kelly<sup>9</sup> tabulated other proposed values for  $A$  and  $B$  and advanced evidence to show that a single conversion equation was inadequate. Barr<sup>10</sup> returned to the subject, and showed that at a temperature of 70° F. the relationship between Redwood seconds and kinematic viscosity could be represented by two equations of the same form, evaluating  $v/T$  in centistokes per second.

$$\frac{v}{T} = A - \frac{B}{T^2}$$

where

$$A = 0.264, B = 189 \text{ when } T \text{ is less than 91 seconds ;}$$

and

$$A = 0.247, B = 50 \text{ when } T \text{ is greater than 91 seconds.}$$

Garner, Broom, and Taylor<sup>11</sup> made the very valuable contribution that the relationship at the usual temperatures of 70° F., 140° F., and 200° F. can be expressed by equations of the general type :

$$v = \frac{T}{10} \times k^{(1 - A/T^n)}$$

This type of equation<sup>12</sup> had previously been proposed for conversion of Saybolt seconds into kinematic units. In 1936 the I.P.T. published its Viscosity Nomogram<sup>13</sup> based on these results. It gave the first authoritative relationship between Redwood seconds and kinematic viscosity with a probable accuracy of 0.5 per cent.

Using this Nomogram the maximum errors that should occur in arriving at kinematic viscosity by means of an N.P.L. certified No. 1 Redwood instrument may therefore be  $\pm 1.5$  per cent. at 100° F., and  $\pm 2.5$  per cent. at 210° F. when the efflux time is about 50 seconds, but if the time is about 100 seconds, the error at this temperature would be reduced to  $\pm 1.5$  per cent. For the calculation of viscosity index such errors would cause too much deviation. Even assuming that the probable error is two-thirds of the



maximum error, considerable deviations would still be possible. It should be acknowledged that large errors in V.I. are less likely to occur if the same N.P.L. certified Redwood instrument is used for determining the viscosities at both temperatures.

From these considerations it appears that the Redwood viscometer does not give the requisite degree of accuracy for the calculation of V.I., except perhaps for oils of relatively high viscosity.

An effort was made in 1923 by the British Standards Institution to replace the Redwood viscometer by U-tube viscometers, through the publication of a British Standard Specification. A second edition was published in 1929 and a third in 1937,<sup>14</sup> so there had been ample opportunity for study and criticism of the design and method. The precision of the method as a whole is not clearly stated by the standardizing authority, but the effect of various errors on the time of flow of liquids in individual viscometers is discussed. An attempt has been made below to summarize and assess these errors.

1. *Dimensions.*—These are such that within the degree of accuracy (0.5 per cent.) and above the minimum viscosity for which the instrument is designed, the time of flow of a liquid will be proportional to its kinematic viscosity. Using the approximate corrections for kinetic energy provided, no error due to this cause should exceed 0.1 per cent.

2. *Filling.*—Provided that the viscometer is filled to within 0.2 mm. of the appropriate mark on the wide tube, no error exceeding 0.5 per cent. should occur. If liquids which have the same surface tension are compared (such as petroleum products), then the filling error should not cause an error of more than  $\pm 0.1$  per cent.

3. *Deviation from the Vertical.*—The capillary of the viscometer should be within 1 degree of the vertical in any plane. The error in time of flow due to a deviation of 1 degree should not exceed  $\pm 0.2$  per cent.

4. *Temperature Control.*—This should be such that three estimations of the time of flow do not differ by more than 1 per cent. If the mean of these readings is taken, this error should not exceed  $\pm 0.3$  per cent. This does not take into account any inaccuracy in the thermometer.

5. *Timing.*—The stop-watch should be reliable, reading to one-fifth of a second; it should be checked frequently, and should be fully wound before use. No error is indicated by the specifications for timing. For the type of stop-watch indicated an error of  $\pm 0.15$  per cent. may be considered reasonable.

6. *Calibration.*—This is carried out using one or more of three standards: distilled water, 40 per cent. sucrose solution, and 60 per cent. sucrose solution, depending on the size of the viscometer to be calibrated. "Stepping up" is allowed. The constant obtained is considered to be independent of temperature, as it increases by less than 0.1 per cent. between 0° and 100° C. No indication is given of the probable accuracy of this constant.

From these considerations, and assuming no error in the calibration constants of the viscometers, it should be possible to measure viscosity by this standardized method with a maximum error of  $\pm 0.85$  per cent. Even if a probable error of  $\pm 0.55$  per cent. is assumed, the effect on the estimation of V.I. is considerable, as shown in Table I.

Many operators have found that differences are encountered when



determining the kinematic viscosity of the same oil in different B.S.S. viscometers, all of which have been calibrated by the N.P.L. As these discrepancies occur in purely comparative work, in which many of the errors assessed above can be practically eliminated, further thought must be given to the subject. The phenomena are particularly noticeable when determining viscosity with a No. 2 B.S.S. viscometer which has been calibrated with 40 per cent. sucrose solution and with a No. 3 B.S.S. viscometer calibrated with 60 per cent. sucrose solution. Steiner,<sup>15</sup> working with four B.S.S. viscometers, the constants of which had been determined by the N.P.L., found the variations shown in Table II. He thus found an overall difference

TABLE II.

B.S.S. Visco- meter No.	Difference, per cent.
2	+0.52
3a	0.00
3b	+0.24
3c	-0.21

of 0.73 per cent. Another worker arrived at a figure of 0.6 per cent. when working with B.S.S. viscometers at his disposal.

When the viscosity of an oil was estimated by us in a No. 2 and in a No. 3 B.S.S. viscometer simultaneously, at the same temperature, a better correlation was found. These results are shown in Table III.

TABLE III.

B.S.S. Visco- meter No.	Filling No.	Time of flow, seconds.	C (by N.P.L.)	Viscosity, centistokes.		
3(d)	1	195.5	0.3420	66.82		
		195.4				
		195.6				
	2	195.5				
		195.4				
		195.6				
		average				
		195.5				
2(b)	3	1406.2	0.04768	67.06		
		1406.6				
		1406.4				
	4	1407.0				
		1406.0				
		1406.2				
					average	
					1406.4	

Difference 0.36 per cent.

Barr<sup>10</sup> had previously shown that the calibration constant of a particular No. 3 tube derived from observations with 60 per cent. sucrose agreed within 0.3 per cent. with the constants deduced by means of (a) oils whose absolute viscosity had been measured in a capillary tube viscometer of accurately known dimensions, (b) stepping up from No. 1 via No. 2 using Bingham's value for water, (c) stepping down from the viscosity of a syrup-



glycerol mixture determined by the falling-sphere method. Unfortunately Barr has not given any comparative figures obtained by the use of 40 per cent. sucrose.

Steiner<sup>15</sup> has shown also that the calibration constant of an individual viscometer may vary with the time of flow, but he does not claim that this is common to all B.S.S. viscometers. For his standard he selected a No. 3 B.S.S. viscometer whose calibration constant was reliable throughout its range when compared with a viscometer of different design. For the information of the authors he compared his standard with their standard No. 3 B.S.S. viscometer, which was N.P.L. certified, and found that in some parts of their range the "calibration constant" of the authors' viscometer varied by 0.3 per cent. From these investigations the following may be deduced:—

1. The use of calibration constants derived from 60 per cent. sucrose solution may result in differences in viscosity of 0.3 per cent.
2. Calibration constants based on 60 per cent. and 40 per cent. sucrose solution respectively sometimes vary by 0.7 per cent.
3. Calibration constants may vary within the range of the instrument.
4. An appreciable discrepancy may exist between a No. 1° and a No. 2 B.S.S. viscometer if the calibrations are made with water and 40 per cent. sucrose respectively.

In Table IV are the results,<sup>16</sup> published in 1938, of the viscosity determinations on the same oil by five different laboratories of the highest integrity

TABLE IV.

N.P.L.	. . . . .	Britain	20.98 cp
P.T.R.	. . . . .	Germany	20.54
B.S.	. . . . .	U.S.A.	21.00
E.N.S.P.	. . . . .	France	21.63
B.P.M.	. . . . .	Holland	20.98

and skill. From the status of these laboratories it is safe to assume that they would select the method in which they had the utmost faith. The maximum difference between these results is 5.3 per cent. The inference to be drawn is that there is a definite need for international standardization. This was actually attempted by calling a meeting of the International Standards Association's Sub-Committee on the Viscosity of Petroleum Products in January, 1939, but no results were obtained from that meeting, owing to the war. Between the British, American, and Dutch laboratories there is a difference of only 0.09 per cent. Whether these three laboratories all used B.S.S. viscometers is not recorded in the published records, but the commendable closeness of the results suggests that instruments of the same basic type were used. The German laboratory may have used the suspended-level viscometer, and the French the Vogel-Ossag. The primary standard employed as the basis for the measurements is not recorded, but had all the laboratories taken values for water ranging from 1.0064 to 1.007 centistokes at 20° C. there would be insufficient variation to account



for the differences shown in the results. If these assumptions are correct, it follows that at least two of the viscometers were subject to errors that had not been correctly assessed.

Consideration may now be given to Table V, which shows some results on the same seven samples of oil, obtained in 1941 by four reputable laboratories in the U.S.A. while investigating the relationship between kinematic viscosity and Saybolt Furol viscosity for the A.S.T.M.<sup>17</sup>

The type of viscometer used is not stated, but it is reasonable to expect that it was of the Fenske or suspended-level variety, since at that time the A.S.T.M. was virtually committed to these two types.

TABLE V.

Sample.	Kinematic viscosity at 122° F.				Maximum difference, per cent.
	1.	2.	3.	8.	
A . . .	662.0	660.9	657.4	654.7	1.11
B . . .	414.5	415.7	413.5	411.7	0.97
C . . .	225.0	225.2	224.6	223.5	0.76
D . . .	145.0	145.0	145.0	143.6	0.97
E . . .	93.8	93.9	93.3	93.4	0.64
F . . .	64.5	64.6	64.1	64.1	0.78
G . . .	47.7	47.7	47.6	47.4	0.63

Both the Fenske and the suspended-level viscometers have been very fully described in the literature. It is perhaps sufficient to indicate their main points of interest, so that comparison may be made with the B.S.S. instruments. Suspended-level viscometers obviate any error due to filling, since the effective head of liquid depends only on the height of the liquid from a point in the suspended level, and does not depend at all on the height of the liquid in the other limb of the viscometer. Any error due to misalignment is reduced by placing the suspended level directly under the upper bulb. This type of viscometer avoids any bottom-bulb errors, but, on the other hand, top-bulb errors are not compensated in the lower bulb. In the suspended-level types with spherical shoulder, differences due to surface tension in the liquids examined are eliminated.

With the Fenske viscometer a definite volume of liquid between fixed temperature limits is charged to the instrument, so that the same weight of liquid is used for viscosity determinations on the same sample at different temperatures. This procedure necessitates the use of different calibration constants for the different operating temperatures, and has the disadvantage that once the instrument is charged, the operator cannot check the volume of liquid. The error due to misalignment is reduced by placing the upper bulbs directly over the lower, which is made comparatively large to reduce errors due to incomplete drainage.

It is stated in the A.S.T.M. tentative method<sup>2</sup> that any viscometer employed shall be capable of measuring viscosity with an error not greater than  $\pm 0.2$  per cent. within its viscosity range. How, then, can this statement be correlated with the maximum differences in viscosity shown in Table V? The explanation, at least in part, may lie in the method used for calibrating the viscometers. Except in the case of the suspended-



level viscometers with capillaries less than 0.75 mm. in diameter, the calibration of which is based on the primary standard pure distilled water, all calibrations are based on oil samples. These oil samples are secondary standards, and may be obtained from the A.P.I. or the Bureau of Standards. The determination of the viscosity of an unknown oil therefore depends not on a single viscosity measurement, but on three separate and distinct operations—namely, the determination of the viscosity of the secondary standard by the standardizing authority, the calibration of a viscometer using the secondary standard, and the determination of the viscosity of the unknown oil using this calibrated viscometer. If it is assumed that standard oil from the same bulk is supplied to two laboratories, it is possible for their results on a given unknown sample to differ by as much as 0.8 per cent., although no error greater than  $\pm 0.2$  per cent. has been made on any single viscosity determination. Any difference that may exist between secondary standards, due to storage or errors in determining the viscosity by the standardizing authority, and any wandering of the calibration constant of a particular viscometer will, of course, further influence the results. The average difference of 0.83 per cent. given in Table V may be said to be quite reasonable and very close to the expected probable error. It may also be inferred that  $\pm 0.2$  per cent. is sufficient margin to cover errors due to temperature, temperature control, timing, alignment, and filling, but is insufficient to cover errors due to calibration with which must be associated errors due to kinetic energy, surface tension, and any idiosyncrasies of a particular viscometer that may cause the calibration constant to vary with the time of flow or temperature.

These recent A.S.T.M. methods are of immediate interest, because it is upon them that the Institute of Petroleum has based its new standard method for determining viscosity in kinematic units.<sup>1</sup> The I.P. methods are, in general, more detailed than the A.S.T.M., and specify the B.S.S. viscometer in addition to the Fenske and suspended-level types with spherical shoulder. The square-shouldered suspended-level viscometer is not allowed. Efforts have been made to minimize some errors. Suitable time-recording devices are described in detail, and thermometers graduated to 0.05° F. are specified as against 0.1° allowed by the A.S.T.M. In addition, the minimum flow time for calibration and referee work is set at 200 seconds, compared with the A.S.T.M. figure of 150 seconds, while for routine work the lower limit is 120 seconds, as opposed to 80 seconds. Suitable constant-temperature baths are described, together with details of the electrical apparatus to employ, but rigid adherence to these details is not called for, possibly because more advanced methods are available. With these advantages of refinement the I.P. state that the apparatus should be capable of measuring viscosity with an error not greater than  $\pm 0.3$  per cent., compared with the A.S.T.M. figure of  $\pm 0.2$  per cent.

The possible error due to calibration of all the various types of viscometers has not been considered in the I.P. Method of Test, hence a few comments on this important aspect are clearly indicated. For suspended-level viscometers pure distilled water is the primary standard, the viscosity being taken as :

1.0068 centistokes at 68° F.  
0.689 centistokes at 100° F.



This standard is directly applicable only to No. 1 viscometers of this type. For B.S.S. viscometers the primary standards are pure distilled water, 40 per cent. sucrose solution, and 60 per cent. sucrose solution, although an observation is made that the figures for the 40 per cent. sucrose solution may require revision. For Fenske viscometers, and also for both the other types, the use of secondary oil standards, as issued by the A.P.I. and the Bureau of Standards, is allowed, in addition to oil samples the viscosity of which have been determined by the N.P.L. The calibration constant determined at 100° F. is considered valid for all other temperatures in

TABLE VI.

Oil.	Filling.	B.S.I. tube No.	Temperature.	Time of flow, seconds.	Viscosity, centi-stokes.	
A.P.I. Beta 42	1	2(b)	210° F.	507.0	24.17	
	2			507.0		
2		507.0				
	2	506.6				
2		507.4				
	2	507.0				
2		average				
	2	507.0				
A.P.I. Beta 42		3	3(f)	100° F.		1124.0
	1122.0					
	1123.0					
	average					
	1123.0					
A.P.I. Alpha 42	1	3(f)	100° F.	182.2 *	62.20	
	2			182.0		
2		182.0				
	2	181.8				
2		182.0				
	2	average				
2		182.0				

\* Flow time rather low for calibration purposes.

the case of the suspended-level types, whilst factors are given to evaluate the calibration constant at various other temperatures in the case of the Fenske.

The order of reproducibility that can be achieved using B.S.S. viscometers calibrated by the N.P.L. with sucrose solution has already been discussed. Table V gives an indication of the reproducibility that may be expected when working with viscometers calibrated from A.P.I. or Bureau of Standards secondary oil standards, while Table VI shows some results obtained by the authors when measuring the viscosity of secondary oil standards in B.S.S. viscometers whose calibration constants have been reported by the N.P.L. Table VII shows the recorded A.P.I. figures and the differences from our own.

The agreement using the No. 3 B.S.S. viscometer is quite good, being well inside the error inherent from its calibration with 60 per cent. sucrose solution. The results obtained at 210° F. using the No. 2 B.S.S. viscometer



are disturbing. They may be due to a combination of factors, although the greatest error can probably be associated with the published data on the 40 per cent. sucrose solution used in the calibration of the No. 2 B.S.S. viscometer.

It may be said with some confidence that the large number of standards now allowed by the I.P. do not give consistent results among themselves, and a discrepancy of anything up to 1 per cent. in the estimation of kinematic viscosity must be expected between two operators when the particular standard to use had not been clearly specified. In lubrication this is usually of minor consequence, but when the calculation of V.I. is involved the discrepancies assume greater significance. It is perhaps fortunate that the accurate determination of viscosity index does not require the true

TABLE VII.

Oil.	Temperature.	A.P.I. recorded viscosity figure, centistokes.	Difference, per cent.
A.P.I. Beta 42 . . . .	210° F.	23.91	1.09
A.P.I. Beta 42 . . . .	100° F.	384.0	nil
A.P.I. Alpha 42 . . . .	100° F.	62.33	0.20

viscosity with a high degree of accuracy. It is sufficient for the purpose of V.I. that viscometers should measure viscosity accurately against an arbitrary standard. For convenience this may coincide with one of the recognized standards, at some definite temperature and for some definite flow time. It must be borne in mind that when the viscosity of a petroleum product is measured at two temperatures to determine the V.I., two viscometers of differing ranges are usually employed. It follows that the calibration constant of these two instruments should be based on the same standard, whether it be 40 per cent. or 60 per cent. sucrose solution, pure distilled water, or one of the secondary standards. It is also advisable to use viscometers of the same basic type, and not a combination of different types of viscometers, some calibrated against one standard and some against another. Steiner<sup>15</sup> suggests the procedure of selecting a viscometer whose calibration constant is probably reliable within the whole range of the instrument, and regarding this instrument as the laboratory standard, at least for V.I. considerations. Other viscometers with the required viscosity range are then carefully compared with the selected viscometer throughout their entire range, and the calibration constants for various flow times are determined. The calibrating procedure would involve much precision work. If this is carefully done the "constants" should be reliable to  $\pm 0.1$  per cent. When these viscometers are used with all the other refinements of the new I.P. method, it should be possible to measure viscosity against the arbitrary standard with a probable error of  $\pm 0.25$  per cent., which is adequate for the calculation of V.I.

The use of charts, or the equations on which they are based, has not been considered in this paper, as it is obvious that they introduce other errors into the calculation of V.I. Apart from the usual charting errors, it is



realized that the very ingenious and useful formulæ which have been suggested are designed to fit experimental results. Such methods, then, are not to be recommended for determining viscosity from which V.I. is to be calculated.

In the U.S.A. there has been an opportunity to obtain experience with the newer methods for determining kinematic viscosity and their application to the calculation of V.I. In this country we are on the threshold of obtaining such experience. Until the sellers and the buyers of oils have thoroughly understood the difficulties, there may be misunderstandings. It is prudent, therefore, to abstain from overstating the accuracy of these methods, and to content ourselves with the conviction that they are superior to those which are being dislodged.

*References.*

- <sup>1</sup> I.P. Serial Designation 71/42 (T).
- <sup>2</sup> A.S.T.M. Designation : D 445-39 T.
- <sup>3</sup> I.P. Serial Designation 73/42.
- <sup>4</sup> Dean and Davis, *Chem. and Met. Eng.*, 1929, **36**, 618.
- <sup>5</sup> Dean, Bauer, and Berglund, *Industr. Engng Chem.*, 1940, **32**, 102.
- <sup>6</sup> I.P. Serial Designation 70/42.
- <sup>7</sup> *Petroleum World*, 1913, **10**, 268, 324.
- <sup>8</sup> R. and M. of Aeronautical Research Committee, No. 906, 1923.
- <sup>9</sup> *Physics*, 1933, **4**, 97.
- <sup>10</sup> *J. Inst. Pet.*, 1936, **22**, 1.
- <sup>11</sup> *J. Inst. Pet.*, 1936, **22**, 11.
- <sup>12</sup> *Industr. Engng Chem.*, 1935, **27**, 977.
- <sup>13</sup> *J. Inst. Pet.*, 1936, **22**, 21.
- <sup>14</sup> B.S.S. No. 188, 1937.
- <sup>15</sup> Private communication.
- <sup>16</sup> Second Report on Viscosity and Plasticity, 1938. Prepared by the Committee for the Study of Viscosity of the Academy of Sciences at Amsterdam.
- <sup>17</sup> A.S.T.M. Standards on Petroleum Products and Lubricants, 1941, 2 pp.

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## THE INSTITUTE OF PETROLEUM.

A MEETING of the Institute of Petroleum was held at the Junior Institution of Engineers, 39 Victoria Street, London, S.W.1, on Friday, 29th January, 1943, at 4 p.m. In the absence of the PRESIDENT, the Chair was occupied by Dr. F. H. Garner.

The following paper was read by Mr. J. C. Cragg :—

“Viscosity Measurement and Viscosity Index.” By J. C. Cragg, B.Sc., A.I.C., and E. A. Evans (Member of Council) (see pp. 99–109).

### DISCUSSION.

THE CHAIRMAN said that the paper was a very interesting one and should serve as a valuable focus for discussion on the question of the accuracy of viscosity measurements.

Before the discussion began it would be interesting to consider the way in which the requirements of accuracy were stated in the I.P. standard method, first with regard to scope, and then with regard to the apparatus. The statement under the latter head was as follows: “The apparatus shall be of the capillary type, and shall be capable, with proper manipulation, of measuring viscosity with an error not greater than 0.3 per cent. within the viscosity range with which it is to be used”; in other words, the figure might be either 0.3 per cent. over or 0.3 per cent. under the true viscosity, and he thought that was on the high side. The error in the procedure was again to be not more than 0.3 per cent. Therefore, if one happened to have an instrument which was 0.3 per cent. out, it was possible to have an additional error of 0.3 per cent., so that the result obtained could be 0.6 per cent. too high or 0.6 per cent. too low, a maximum error of 1.2 per cent., as compared with the A.S.T.M. figure of 0.8 per cent. However, it was the probable error that should really be considered rather than the maximum error, and it could be assumed that the probable error would be about half the maximum error, so that the viscosity determined according to the I.P. standard method should be within 0.3 per cent. of the true viscosity.

With regard to Table V, Sample A, which had the highest viscosity, had a Saybolt Furol viscosity of 310, so that it was possible that that oil was not a lubricating oil, but a fuel oil of the Bunker C type; the deviation in the kinematic viscosity was twice the deviation in the Saybolt Furol viscosity, which was rather unusual, so perhaps it was justifiable to leave Sample A out of consideration.

Another point was that the Kinematic Viscosity of laboratory No. 8 was low compared with the other figures. If these values were omitted, the accuracy would be very considerably improved.

Mr. Cragg had referred to the work of the International Standards Association. That work had involved the use of a series of U-tube viscometers calibrated by the N.P.L., which were circulated to all the standardizing authorities. The times of those tubes were first determined by the N.P.L. on two oils; then the viscometers were sent out to the various countries, with three other oils, and the times were run together with the kinematic viscosities determined by the standard method in use in the particular country concerned. The results, unfortunately, had not been completed, owing to the war, but a number of results had been obtained, which Dr. Barr sent to him last week, and he proposed to put them on the blackboard at a later stage of the meeting.

PROFESSOR GARNER subsequently wrote :—

The point has been made in the paper that the accuracy required in viscosity determination for actual lubricating-oil requirements is much less than that specified in Standard Methods of Test but the accuracy limits in viscosity determinations are determined by specification requirements. It is generally agreed that the figures



quoted in specifications should be taken as absolute—that is, that it is not permissible to include experimental errors and therefore if the viscosity requirements specify figure  $A$  and the experimental error is  $B$ , then the viscosity which actually has to be met is  $A + B$  or  $A - B$ , depending on whether  $A$  is a maximum or minimum viscosity. Where there is no great difficulty from the supply point of view in meeting the viscosity requirement, very great accuracy is not required in laboratory determinations, and thus the figure  $B$  can be made much larger than is specified in the Institute of Petroleum Standard Method, still making sure that the absolute requirement of the specification—namely,  $A$ —is met. On the other hand, in the case of high-viscosity-index oils, it may be desirable and necessary for the supplier to have available viscosity methods and apparatus which enable him to meet the specification requirements with the minimum of error, and for this purpose  $B$  should be as low as possible—for example, as specified in the A.S.T.M. procedure,  $\pm 0.2$  per cent.

The second point concerns the accuracy of the Redwood Viscometer. The Institute of Petroleum Standardization Sub-committee dealing with the Redwood Viscometer some years ago spent much time trying to improve the possible accuracy of the instrument, as, for example, by increasing the accuracy with which the orifice tube is drilled, and it considered for this purpose the use of stainless steel. A Redwood instrument as an absolute viscometer has two fundamental faults: (1) A shortness of the capillary tube and the difficulty of manufacture with reasonably fine accuracy; and (2) the fact that the jet is not maintained and cannot be maintained at the temperature of test, since the bottom plate of the part of the viscometer bath containing the oil is exposed to the atmospheric temperature.

Thus, as laid down in the Standard Methods of Test, it is not advisable to use times in the Redwood viscometer for conversion to centistokes, as the accuracy expected in centistokes by the I.P. method is greater than that which can be obtained with the Redwood instrument.

DR. G. BARR, referring to the early history of the U-tube viscometers, said that when he first had some viscosities to measure, all that he could buy in the way of U-tube viscometers from the laboratory furnishers was an Ostwald viscometer, and he had no idea what the capillary would be, the time of flow, or anything else. He looked at a number of different types and patterns of viscometers that had been got out, and it appeared that the type which had now been adopted by the British Standards Institution offered conveniences, in that one did not need a pipette for filling it and did not have to weigh it when it was filled. He had calibrated a series of those tubes, as Mr. Cragg had pointed out, by comparison with an absolute viscometer of which the capillaries had been measured fairly carefully, by comparison with the then accepted value for water and by stepping down, assuming Stokes' law, from the viscosity of a certain viscous syrup of about 80 poises. (In those days poises were used, and not stokes.) When he had completed the calibration he had compared the figure with that given by Bingham for a 60 per cent. solution of sugar of extraordinarily high purity. He himself had not got any of this grade, so he used A.R. sugar. He worked out a curve or formula expressing Bingham's results, and assumed that he could calculate the viscosity of an approximately 60 per cent. sugar solution from the density. That formula was subsequently adopted in the first B.S.I. specification, and he might remark that, over the range in which he had used it, which he thought covered most of the range which was given in the specification, and with all the different brands or types of sugar which he had used, he had always obtained concordance within about 0.3 per cent. At that time 0.3 per cent. was considered extremely good. The only time he got a 60 per cent. sugar solution which did not agree was when he had some super-pure sugar, which he eventually discovered had a rather strong smell of alcohol. As Mr. Cragg had remarked, he did not then check so exhaustively the viscosity of a 40 per cent. sugar solution, and he had not done so since. Having regard to the difficulty of preparing and measuring a solution of 60 per cent. and measuring its viscosity without picking up water, he thought at the time that if he could repeat the 60 per cent. sugar solution, the 40 per cent. sugar solution must be right, and he left it at that, and the value based on Bingham's results was incorporated as a standard for the calibration of No. 2 viscometers in the B.S.I. tubes. Since then he had reason to believe that that standard differed by  $\frac{1}{2}$  per cent. or so (sometimes it was 0 and sometimes it was 1) from the standard obtained with 60 per cent. sugar,



and of recent years, when asked to calibrate a viscometer or to determine the viscosity of an oil, he had always taken great care to state in his report whether it depended on a viscosity of 60 per cent. or a viscosity of 40 per cent. It was obvious that for the viscosity index determinations one must have only one of the two. He did not think it mattered very much which it was, but, since the 60 per cent. sugar solution had to be used for the higher and it agreed with other determinations, if one used a sugar solution at all the 60 per cent. sugar solution was the obvious one to use.

There was a more disturbing fact, to which Dr. Steiner had directed attention, and which had also been brought out in the calibration of the thirty-two viscometers used for the international calibration to which Dr. Garner had referred—viz., that the calibration constant could on occasion vary with the time of flow. From the series of I.P. viscometers to which he had referred he had selected the extreme examples. He had compared an oil which gave 909 seconds in the one that he kept at the Laboratory with the flow of that oil in each of the other viscometers, and he had done the same with another oil of 113 seconds. The worst of the No. 2 viscometers gave a ratio of time of flow of 0.715 for the oil of 909 seconds, and 0.7130—i.e., differing by 2 in 700—for the less viscous oil of 113 seconds. That result was not too bad, especially as the No. 2 viscometer was the one which had been giving most trouble. With the No. 3 viscometers, however, the ratio changed from 0.881 to 0.886 when the time varied from 160 to 480 seconds, so there was the  $\frac{1}{2}$  per cent., or a little more, straightaway.

He was not prepared to say what that variation could be due to, but one obvious suggestion was that, when one had filled to the filling mark and the oil had been sucked up into the timing bulb, there was a certain amount of oil left in the lower bulb, the quantity of which might vary with the viscosity of the oil. That was all very well, but what one was concerned with was not that quantity of oil, which it was true would make a difference in the viscosity, but the variations in the quantity of oil between different viscometers, and, if the variation of  $\frac{1}{2}$  per cent. or so was to be assigned to the oil which was left behind in the lower bulb, the quantities of the oil left behind must vary between different viscometers, and not only in the same viscometer.

In Table IV the authors had listed the results of some oil determinations of about twenty-one centipoises, and said that whether the three laboratories which agreed—namely, the British, American, and Dutch—used B.S.S. viscometers was not recorded. In that connection he would say that the Bureau of Standards almost invariably used a Bingham type of viscometer, with applied pressure; the British N.P.L. used a B.S.I. instrument, and he was not sure what the Dutch used. He was almost certain that the German laboratory would not have used a suspended-level viscometer, which was due to Ubbelohde, because Dr. Erk had a special absolute viscometer of his own at the Reichsanstalt, and the oil determinations there were presumably based on that absolute viscometer. He might say that Dr. Erk's figures had tended to disagree with those of the other countries in other determinations. A pair of U-tubes had been circulated about 1934 for calibration by different countries, and in that case also the German laboratory was different. With regard to the French determinations, the *Ecole Nationale* used to use a Louis viscometer of an absolute type, and in the comparisons to which he had just referred there was a very serious discrepancy between the French determinations and the remainder.

He had not studied the question of the correct method of assessing possible errors in results if an error of 0.2 per cent. was made in three different connections. It was true that the error might be the sum of all of them, but he thought that in general, as Dr. Garner had remarked, the probable error was not really the whole sum.

DR. N. ANFILOGOFF said that in listening to the paper he had been constantly reminded of the story of the student who took the result of a mathematical calculation to his professor, the figure he had arrived at being 0.525643, and the professor looked at the figure and said: "Well, I do not know about the 643, but the 525 is certainly wrong."

He entirely agreed with what the authors had said in the last paragraph of their paper, that there was a tendency to overdo viscosity index, particularly in connection with commercial specifications. He thought it was a kindly warning that the authors had given, lest one day it might be necessary to meet a specification with a minimum V.I. of 64 and a maximum V.I. of 65.

There was one point to which he would like to refer in connection with Table IV. In 1938 he had sent an identical sample to the French naval laboratories at Toulon,



Paris, and Rouen, and to laboratories at Antwerp, Berlin, and Strasburg, and also to the N.P.L., and the difference in viscosity was of the order of 6 or 7 per cent. It was interesting at the time to note that his own laboratories showed a very much smaller difference between themselves than the three standard laboratories, and also that they were closer to the N.P.L. than to any of the Continental laboratories. Their instruments were based on American standard calibration. It was interesting to see that the figure which Dr. Barr had obtained at the N.P.L., and the figure obtained at the Bureau of Standards had only  $\frac{1}{10}$  of 1 per cent. difference between them. Dr. Barr's results really corroborated what he had found, that the English figures were very different from those of the French and Germans. He had found exactly the same thing in the sulphur determination of a lubricating oil.

DR. BROOM said he would like first of all to congratulate the authors on their thought-provoking paper, with much of which he was in complete agreement.

When the tendency to use kinematic viscosity grew up among petroleum technologists they probably thought that by abandoning, or partly abandoning, the use of the Redwood viscometer and changing over to kinematic viscosity, they were giving themselves a better tool, which was correct; but perhaps they also assumed that they would obtain better results and do a better job of work just because they were using a better tool, and that, of course, did not follow at all. In general, the U-tube type of viscometer would give better results, but it must be recognized that the same degree of painstaking work was necessary in order to obtain accurate results; in other words, it was necessary to observe all the details about temperature, and so on, just as carefully as with the Redwood instrument. The point was that, if that was done, then a better and more correct result was obtained than with the Redwood viscometer.

With regard to the question of sugar solution, he had tried at different times to calibrate No. 2 B.S.I. U-tube viscometers with 40 per cent. sugar solution, and he had compared the answer with what he obtained when he stepped up from a viscometer suitable for the use of water. He always found a difference in the two methods of approximately  $\frac{1}{2}$  per cent. At first he thought that that was probably his fault, that he had made some mistake, but after it had happened more than twice he began to think that there might be something wrong with the 40 per cent. sugar solution, and he believed that had been the experience of several people. Quite apart from the question whether the 40 per cent. sugar solution or the 60 per cent. sugar solution was right or wrong as a basic standard, he had always thought that its use was not a good method to stipulate in a standard methods book, because the tendency would then be to assume that it was a method that could be applied in almost any laboratory and the right answer obtained. Dr. Barr had referred to the difficulties experienced in working with a sugar solution, and they were very considerable, because the solution was hygroscopic, and it was necessary to take fairly elaborate measures to ensure that the particular specimen the density of which was being measured remained the same when in the viscometer and while it was being used. He did not think that was a simple sort of thing for a routine laboratory to do. It might be that a sugar solution could be used satisfactorily in laboratories such as those mentioned in Table IV of the paper; for instance, the N.P.L.

In considering the question of accuracy in general, he had always regarded it as being composed of three parts. Firstly, the question of the basic accuracy came down in the end to an absolute measurement made on a pure chemical liquid, a liquid of defined and reproducible properties, of which pure water was probably the simplest example. Unfortunately it possessed a viscosity which was far too low for most of the purposes in question, and, if a suitable hydrocarbon could be found which had a viscosity more approaching that of a lubricating oil and well-defined properties which could be checked, that would be a considerable help. Would all the various instruments which measured absolute viscosity in the various standardizing laboratories give the same answer when measuring the same liquid? He was referring now to the absolute instrument, not to any instrument calibrated from it. In other words, what was the viscosity of water? Was everyone agreed on the viscosity of water at 20° C.? If not, it would not be possible, as far as he could see, to obtain agreement between the various standardizing laboratories. If there was agreement on that basic standard, the second question was whether the subsidiary instruments, such as the Ubbelohde, the B.S.I., and so on, when they were calibrated or the smallest one in each range was calibrated with water, would still give, over the whole range, correct answers when



more viscous liquids were used. Was the design of each of those viscometers satisfactory, so that they would give the same constants over long times of flow and short ones, due allowance being made for the kinetic energy errors, and so on? There was the third error, which he supposed might be described as an operational error, and that embraced all the experimental details, such as making sure that the temperature was right and that the viscometer was upright in the bath, and all the operational details of a particular determination. Having regard to these three types of error, his own opinion was that, at the present time, it would be optimistic to expect an absolute error in a viscosity determination of less than  $\pm \frac{1}{3}$  of 1 per cent., although the operational error was much less, probably  $\pm \frac{1}{4}$  per cent.

With regard to the estimation of viscosity index based on viscosity measurements, he thought that the authors really accepted the viscosity index principle and its use generally; he did not get the impression that they were opposed to its use. It should be realized, however, that in the case of the lighter oils the viscosity-index system was certainly not uniform. Consider, for instance, the theoretical highest maximum viscosity index of an oil, when the viscosity did not vary at all, but was the same at 210° F. as at 100° F. The viscosity index could be worked out for oils of different viscosities, and in the case of the thinner oils the figure was very high, being 366 for an oil of 2 cs., but as the viscosity of the oil increased it flattened out at about 150 for an oil of 50 cs. viscosity. That must be an indication that the viscosity-index scale itself was not linear; in other words, an error of 5 viscosity units would not mean the same on any part of the scale.

Mr. J. CANTOR, referring to the question of calibration, said it had been shown that in order to obtain results of the desired reproducibility, it was essential to have a single calibrating liquid only. It was necessary to consider which substance had suitable characteristics so that it should be selected as the sole standard. Considering the existent standards first, water had certain disadvantages. First of all it was of low viscosity and it gave an extremely short time of run when it was used for calibrating viscometers. For instance, in the case of the No. 1 Ubbelohde the times of run were about 100 seconds at 20° C. and about 68 seconds at 100° F., which were much too short. Sugar solutions presented difficulties which had already been mentioned. First, they were hygroscopic, and, secondly, it was difficult to measure the density to five decimal places, which was necessary when sugar solutions were used. Furthermore, there was the doubt with regard to the figures for the 40 per cent. sugar solutions. In addition, there was an inherent difficulty about all aqueous media if they were used for calibrating viscometers which were then going to be used for the determination of oil viscosity. For example, the surface tension of water and of sugar solutions was of the order of 75 dynes per centimetre, whereas in the case of oil it was about 30 dynes per centimetre. That led to considerable errors, for which the A.S.T.M. attempts to compensate. In the case of the calibration of the square-shouldered Ubbelohde, the A.S.T.M. determines the constant of the instrument with water and multiplies that constant by 1.0035 in order to get the correct constant for oil-viscosity measurements. In the case of the spherical-shouldered Ubbelohde, the effect of the surface-tension differences was supposed to be eliminated, but there seemed to be some doubt about that. It had been stated by Cannon and Fenske, he believed, that the error in going from water to oil with the usual capillary viscometers was of the order of 0.3 per cent. to 0.5 per cent., and in general it was obviously difficult to assess accurately the necessary correction.

Other liquids had been suggested as primary standards; for example, Steiner had suggested aniline, which had certain advantages. Its surface tension was much nearer to that of oil, being 44 dynes per centimetre, and it had a kinematic viscosity of 4.5 centistokes at 20° C., which gave sufficiently long times of run to diminish considerably the timing errors. Slight oxidation apparently did not affect the viscosity of aniline much, and it seemed to be a promising material.

He would like to suggest another material for consideration, namely, cetane, the diesel-engine standard. Cetane (*n*-hexadecane) had a viscosity of about 4.5 centistokes at 20° C. or 68° F. It had a melting point of 65° F., and the temperature of 68° F. might be too close to that for safety, but the times of run would still be sufficiently long at 77° F. and at 100° F. for the timing errors to be small. Its surface tension was obviously of the correct order. There would be no difficulty in using for the measurement of oil viscosity a viscometer which was calibrated with cetane. It



would be necessary, of course, to establish that the different batches of engine-standard cetane all had the same viscosity or could be converted to the same viscosity by means of a simple treatment. A number of official laboratories would have to determine the viscosity of cetane with extreme accuracy and to agree upon a figure, and then cetane could be used for the standardization of the No. 1 Übbelohde, the No. 50 Fenske, and the Nos. 0 and 1 U-tube viscometers. The other viscometers in each series could then be calibrated by means of a very careful stopping-up process. All the viscometers would then be based upon a single substance and there should be interconsistency in calibration constants. Possibly some other substance might be found to be more convenient, but cetane appeared to be the only stable hydrocarbon which was available and which had suitable viscosity characteristics.

There was an alternative method of clearing up the calibration difficulty, which was less attractive theoretically—namely, to abolish primary standards altogether and to calibrate viscometers with secondary oil standards only. These should be issued by a single authority, such as the N.P.L. That single authority would measure the viscosity of these oil standards with a single master instrument, or with instruments whose constants had been obtained on the basis of the master. All viscometers in use would then refer back to that master instrument, leading to interconsistency in their calibration constants.

Some such scheme as the above would seem to be necessary to eliminate the existent confusion in the field of viscometer calibration.

MR. J. ROMNEY, referring to the question of the possible error in viscosity determination, said it was mentioned in the paper that the error due to one degree deviation from the vertical should not exceed  $\pm 0.2$  per cent. In the case of temperature measurement the temperature might be too high or too low, giving a deviation in either direction, but in the case of deviation from the vertical was not the error in one direction only? Did it seriously matter, for all tubes, whether the tube leaned over in one direction or the other? The error was not a plus or minus figure. It had been worked out that the possible error on the basis of the B.S.I. tubes was the sum of the various errors, and that was 0.85 per cent. It was also suggested that the probable error was likely to be about half that, which was fairly reasonable, but it should be remembered that the errors due to different points might cancel one another out, and therefore, on this basis alone, the probable error became half. Having regard to the fact also that the probable errors due to each point were themselves likely to be 50 per cent. of the possible errors, he thought it was reasonable to assume that the probable error was about one-quarter of the possible total error, *i.e.*,  $\pm 0.2$  per cent., which was in line with the 0.3 per cent. mentioned in the I.P. Standard Methods for Testing. At the same time it was important to realize that one could not work out possible errors by calculating theoretical errors due to the different points. The possible errors were those which did actually arise. A series of tables was given in the paper showing the results obtained by different laboratories, and it must be remembered that these were laboratories which were examining oils under conditions in which they knew that the results would be published, so that extreme care must have been taken in the determinations, yet the maximum deviations were a good deal larger than would suggest the accuracy which it was claimed that it should be possible to obtain. Therefore under present conditions too much reliance should not be placed on viscosity determinations to within very close limits. The effect on the viscosity index had been mentioned. The viscosity index was undoubtedly a very useful tool, but did the deviations which resulted from viscosity errors matter very seriously? He regarded the viscosity index as giving a general idea of the viscosity temperature curve of an oil, and he did not suppose that two or three points either way, or even five points, mattered very much. Dr. Anflogoff was anxious about the appearance in specifications of too narrow limits and referred to a maximum V.I. of 65 and a minimum V.I. of 64, but personally he did not think that anyone would specify a maximum V.I.; it would be only a minimum. It was well known that an oil with a V.I. of 70 was a good deal better on the viscosity temperature curve than oils of 35, and the effects of viscosity errors on viscosity index were probably not very important, outside specification work.

The viscosity index was based originally on viscosities at 210° F. and 100° F., and he thought it was rather a pity that the temperature of 210° F. had been adopted; it was not a suitable temperature, because it did not allow of water being used as the bath liquid. He thought it would be very useful if somebody in this country could



find time to draw up a table from which could be calculated the viscosity index from viscosities at 200° F. and 100° F. That should be quite feasible; it would involve a good deal of work, but some public-spirited person might be willing to do it. At the moment the temperature of 210° F. had to be used, and he would be interested to have suggestions of a suitable bath liquid for that temperature. The I.P. book mentioned oil, but that would be rather unpleasant when one had to lift the tube out of the bath. Ethylene glycol had been suggested, and he thought that would be a good deal better, but it was almost impossible to obtain it at the present time. He did not know of any other suitable bath liquid.

It was stated in the paper that the reference oils calibrated by the N.P.L. could be used as secondary standards, but that they must be re-checked periodically, and he would be glad to have some information about the changes which had been found to occur in oils in storage in stoppered bottles kept in the dark. He thought it was unlikely that most laboratories would have their reference standards checked as frequently as was suggested in the I.P. book, and he would like to know what would happen if the time between checkings was rather longer than the time mentioned in the book.

It was rather disturbing to learn that the factor for a particular tube might vary with the different times of run, and he would like to have some information about the idiosyncrasies of tubes that caused these changes. If there was anything that one could actually look for, it would be a good deal simpler than having to obtain the factors against the numerous standards that were suggested in the paper.

Mr. ROMNEY subsequently wrote :

Since commenting at the meeting on the calculation of the probable from a series of possible errors, I have taken the opportunity of looking up this subject and of discussing it with mathematicians more expert than I. It seems that the result depends on the interpretation of the term "possible." It is hardly true to say, in the instance under discussion, that the possible errors are those which will never be exceeded. If it is assumed that a possible error is one which will not be exceeded more than once in five times, then reference to a table of the Probability Integral shows that the probable error of a measurement with a possible error of 0.1 is :

$$\frac{0.6745 \times 0.1}{1.2816}$$

The square of the probable error of a sum of quantities is the sum of the squares of the probable errors of the separate quantities. Hence, for the series of possible errors indicated by the authors, the probable error is :

$$= \pm \sqrt{\left\{ \frac{0.6745}{1.2816} \right\}^2 (0.1^2 + 0.1^2 + 0.2^2 + 0.3^2 + 0.15^2)} = \pm 0.2186 \text{ per cent.}$$

If the possible error is one which is likely to be exceeded only once in ten times, the factor becomes :—

$$\frac{0.6745}{1.6449}$$

and the probable error works out at  $\pm 0.1703$  per cent.

Even if the possible errors are those which are as likely to be exceeded as not, the probable error becomes  $\pm 0.4153$  per cent., which is lower than the  $\pm 0.55$  per cent. mentioned in the paper, but it is unlikely that the authors intended this interpretation to be placed on the term "possible error."

This would appear to suggest that errors in the calibration constants are mainly responsible for the relatively large differences between the viscosity figures reported by different laboratories on the same oils.

Mr. J. S. JACKSON thought that ample evidence had been given in the paper and in the discussion to show that the idea that the true viscosity of an oil could be determined within  $\pm 0.2$  per cent. as an ordinary routine operation was a pure myth. If it was a pure myth, he thought something should be done about it and the Institute should take the matter up strongly with the A.S.T.M., because their method, whatever it really meant, gave the impression that with the apparatus in question that degree of



accuracy could be obtained in ordinary commercial laboratories. It had been said that five points on the viscosity index did not make a great deal of difference, but that was not the case if one had to supply an oil of 100 viscosity index and happened to produce one that was 95 by mistake. On the other hand, if the specification called for an oil of 100 K.V.I., it was obviously not feasible to make one of 105 for safety.

With regard to the question of the No. 2 B.S.I. viscometer, an effort had been made to get out of the difficulty by saying that "it is recognized that the figures for the 40 per cent. sucrose solution may require revision." Dr. Barr had also admitted that he had reason to believe that the 40 per cent. sucrose figures might not be very accurate. Personally he submitted that the average man in the average laboratory up and down England was probably not in a position to appreciate this very unfortunate state of affairs, and if he bought a No. 2 N.P.L. certified B.S.I. viscometer, he would naturally expect it to enable him to determine accurately the viscosity of an oil. If, however, it gave him a figure which differed by about 1 per cent. from the result obtained with a No. 3, he might get into serious commercial difficulties. Therefore he would suggest that the Institute should take the matter up with the B.S.I. or the N.P.L., and in the meantime it was suggested that all reference to the B.S.I. viscometers should be deleted from the Institute's Method Book.

MR. E. A. EVANS said that Mr. Jackson had made remarks that he had intended to make. The Chairman had suggested that there was a legal mind behind the paper, and that there had been a good deal of care exercised in the wording of it. Mr. Marlow, who was known to many of the members, gave a series of lectures on Law which he had attended. In those lectures Marlow impressed upon his hearers the great need for thinking as the legal mind thought. Presumably it is to collect the facts in logical sequence, and to assemble them with just sufficient gaps to leave room for an argument. In this paper the authors had purposely left gaps, to invoke argument. Mr. Jackson had seized upon some of the gaps, and had spoken about them. No one, however, had dared to say definitely whether the Americans were using the Saybolt instrument as the basis for viscosity index. It would be useful to have an authoritative statement on this one point, because then we should be in a better position to gauge their accuracy of viscosity index. The essence of this paper was to bring into bold relief that a high degree of accuracy is needed in viscosity determination when an accuracy of a few points is demanded in the calculation of viscosity index. So long as the Saybolt instrument is the preferred instrument in the U.S.A., we need not be too fussy about controlling the viscosity thermostats to plus or minus 0.025° F. Lately some embarrassment has been caused by one of the Government Departments demanding an oil with a particular viscosity index. The embarrassment lay, not in obtaining the correct viscosity index, but in deciding how many points above it it would be necessary to go to ensure meeting the specification. Clearly the answer lay exclusively in the accuracy of the viscometer which should be used. When viscosity index is translated into pence per gallon, and gallons into thousands of gallons, the importance of viscosity measurement becomes a little more obvious. The question which must be decided sooner or later is whether kinematic viscometers shall be used on all occasions when viscosity index is to be calculated. No one seems to be bold enough yet to answer that question; it is one which must be faced if we are expected to state viscosity index to an accuracy of three points. In making this statement he was not referring to the calibration of a particular instrument, or the measurement of the viscosity of a particular oil, but to oils which are being made and sold every day. We are not dealing with an academic question, but one of routine.

THE CHAIRMAN then put on the blackboard the following table, showing the figures he had received from Dr. Barr, to which he had referred in his opening remarks.

	1.	2.	3.
Ubbelohde . . . . .	528.7	17.77	79.00
Fenske . . . . .	527.6	17.76	78.89
Absolute . . . . .	528.5	17.79	79.08
U-tube (I.P. Standard Method) .	527.0	17.70	78.77



The table showed the determination made on three oils in laboratories in America, the last one being the figures determined in this country with the U-tube viscometer. He thought it would be agreed that the accuracy shown in those figures, which were determined under conditions of extreme care, were very good and supported the A.S.T.M. figure of 0.2 per cent. It was interesting to note that the figures obtained in this country were a little low, as in Table IV of the paper, where the N.P.L. figure was a little low as compared with the figure obtained by the Bureau of Standards. All three types of instruments in the I.P. Standard Methods were represented.

It appeared desirable to review the work done for the International Standards Association, and he hoped that at a later date it would be possible to present a paper summarizing all the results. The results were incomplete in the case of the Continental countries, because the samples were circulated only in May or June, 1939, and it was for this reason that no summary had yet been prepared.

Mr. R. F. HURT said that attention had been focused mainly on the calibration of the instruments, but that it seemed to him that the oil also should be considered. Mr. Evans had referred to an oil of Viscosity Index 85, that oil being loaded with 10 per cent. of additive. What effect would that additive have on determinations of kinematic viscosities, and their reproduction by different laboratories? Discrepancies, instead of being due to apparatus, could well be inherent in the oil actually under test.

He had no doubt that all the figures given in the paper had been determined on beautifully bright, clear, moisture-free oils, but how many of the members invariably dealt with oils in that condition? A number were concerned with oils from ships' and shore tanks, or from batches in process of manufacture. Such oils would contain traces of moisture, perhaps contain an unknown additive, or have been heated in some way immediately prior to test.

The details in the I.P. handbook for the Redwood instrument provided for the question of the thermal history of the oil under consideration. It is laid down that it should be heated in a stoppered container for 1 hour at 212° F. in a suitable bath maintained at that temperature. In passing, he wondered how many laboratories, when doing ordinary Redwood viscosities for works control, followed this procedure, and how many took a sample, warmed it in a pan (which according to Redwood details is not permissible), and measured the viscosity at once.

In the older, less accurate Redwood method the advisability of a uniform heat treatment before test is recognized. This is not the case with the newer system. He felt that with regard to kinematic viscosities, there should be some ruling as to a quick preliminary thermal treatment, and so far this has not been given.

Mr. LAPITZKY said that his firm had done a fair amount of calibration of viscometers, and he felt that they could obtain an accuracy to within 0.2 per cent., provided they could get a definite basic standard. That had been their main trouble. They could get a viscometer calibrated accurately provided they had an exact and reliable figure for their standard. They found that the temperature-control difficulty was not so very great, but the difficulty of obtaining suitable interconsistent standards was very considerable.

Mr. LAPITZKY subsequently wrote to suggest :

(a) Removal of the B.S.I. viscometers, and possibly the Fenske viscometer from the I.P. Standard Methods. I know that apart from the question of accuracy many laboratories have experienced difficulties in the use of B.S.I. viscometers, and there is a feeling that these are not really suitable for petroleum work.

(b) The abolition of primary standards, such as water and sugar solutions, the calibration of viscometers to be effected by means of secondary oil standards only. Those oil standards should be obtainable from one authority only—for example, the N.P.L.—who will guarantee their viscosity on the basis of a single master viscometer, the constant of which shall be determined from first principles. This authority might issue sub-master viscometers, calibrated against the master, to other official laboratories, who would then be in a position also to issue secondary oil standards; all these secondary oil standards then being strictly interconsistent.

(c) An alternative scheme would be the establishment of cetane, as suggested by Mr.



Cantor, as a primary standard, provided this material can be obtained in a sufficiently pure state and is stable in storage. The higher viscometers in the usual range would then be calibrated by a careful stepping-up process.

(d) In any case supplies of certified pure cetane and/or of certified secondary oil standards should be available from the I.P. The certified values allocated to these standards should definitely correlate with the oil standards supplied by the A.P.I.

Mr. H. L. WEST said that he would like to take the authors to task on one point. They referred in their paper to the conversion of Redwood units to kinematic units. This should not be done; convert kinematic to Redwood, but not Redwood to kinematic units. (See "Standard Methods for Testing Petroleum," Fourth Edition, 1942; Appendix, p. 383.)

He agreed with the authors that it was possible to obtain considerable variation in the calibration constants with viscosity using B.S.I. tubes. In comparing the same N.P.L. calibrated B.S.I. tube with three different Ubbelohde tubes, he had found that there was quite a large error, according to the time of flow, in the B.S.I. tube, amounting to about 0.7 per cent. with an oil of 152 centistokes, although at 351 centistokes the B.S.I. tube appeared correct. This is illustrated in the figures below.

Ubbelohde Viscometer No.	Viscosity of secondary standard oil (centistokes).	Viscosity of oil according to N.P.L. B.S.I. tube (centistokes).	Calibration constant of Ubbelohde tube.
3/20	—	152.3	0.9221
"	—	269.7	0.9219
"	—	351.3	0.9183
3/21	484.0	—	1.288
"	—	152.3	1.234
"	—	269.7	1.234
"	—	351.3	1.229
3/22	78.99	—	1.002
"	528.6	—	1.004
"	484.0	—	1.001
"	74.02	—	1.002
"	—	152.3	1.008
"	—	269.7	1.005
"	—	351.3	1.001

It will be seen that the calibration constants of the Ubbelohde tubes are constant against the secondary standard oils, whereas against the oils the viscosity of which has been determined in the B.S.I. tube, the constants vary appreciably.

Since the laboratory with which he was associated had had as much experience as anyone in the use of these more or less accurate methods of determining viscosity, he would outline the procedure adopted. In the first place they used the Ubbelohde tube, and made a practice of having one or more tubes in each range carefully calibrated against secondary standard oils, using these as master tubes for calibrating other viscometers for use in the laboratory. Having satisfied themselves that the master tube was as accurate as could be obtained, the other viscometers were calibrated against the master tube, using a range of oils which were run in the master tube at the same time as in the tube being calibrated. By that means he thought they obtained fairly good calibration; also they made sure that the calibration did not vary over the range by more than  $\pm 0.1$  per cent.

With regard to the oil used for calibration, it was necessary to be quite sure that the flow of the oil was satisfactory—*i.e.*, that it is Newtonian. In commercial work he did not think it mattered so much, but for calibration purposes it was a necessity. They made sure that only highly refined oils were used when calibrating against the master tube. So far as temperature control was concerned, they controlled the thermostats used to within 0.03° F. of the temperature of the test.



He maintained that the measurement of viscosity, provided a suitable tube was used, could be carried out with quite good results. He had found that people with no experience whatever who had visited their laboratory, obtained just as good results with the apparatus as those who had been doing such work for years. So much so that he maintained that, if the control was taken out of the hands of the operator, he thought an intelligent monkey should be able to measure viscosity. The only possibility of error was that dirt or other extraneous matter would get into the tube, with the result that an incorrect time of flow would be given.

He thought it would be very useful, particularly to the smaller laboratories, if the Institute could arrange a source of secondary standard oils similar to those available from the U.S.A. through the A.P.I.

DR. G. BARR, replying to some of the remarks made in the discussion, said it was true that the error due to inclination from the vertical was always in one direction for instruments of the Ubbelohde type, but it might be in either direction for the U-tube type.

With regard to the standardizing of the instruments, Dr. Garner had put on the black-board a collection of four results. Could it be assumed that at any rate three of the laboratories in question had used the same standardizing oils to calibrate their viscometers? It was probable that the determinations did not in all cases profess to be absolute determinations, starting from scratch.

THE CHAIRMAN thought that the Bingham viscometer was the only absolute viscometer used.

DR. BARR asked whether the other viscometers were merely calibrated by oils supplied from outside.

THE CHAIRMAN said that he thought the first two would be considered dependent on the third, but the fourth would be a combination of the first three, the American standard and the N.P.L. standard.

DR. BARR said the point he wished to make was that uniformity of standards would obviously increase reproducibility as between one country and another. It was rather difficult, he imagined, to interchange oil samples between the United States and this country and to rely on the viscosity of that oil being constant for the requisite number of months or years.

The suggestion to use aniline as a primary substance was an interesting one, because it might overcome some of the difficulties which occurred in the stepping up from water.

With regard to stepping up from the assumed known viscosity of water to the viscosity of an oil by a series of instruments, he might mention that in the investigations to which he had referred earlier in the meeting the U-tubes which were used were not British Standard tubes, because there were none. They were merely tubes which he made up with what he thought would be suitable capillaries and which he thought would give similar times of flow, and they probably differed in a number of respects from the British Standard tubes. In that case he had managed to get concordance between the viscosity in the No. 3 range and the viscosity of water by stepping up, and he might mention that when he first discovered the discrepancy in the 40 per cent. sugar solution, he thought the simple and obvious method of getting over it would be to step up from water, as he had done before. He therefore did that, with a brand-new set of tubes, but got no confirmation at all. He did not know then at what stage in his stepping up the error had crept in, and he still did not know, because it seemed to him that, in any series in which stepping up was done, if the calibration constants could vary as they had been shown to do in some cases, the error was bound to occur. If the variations were due to drainage and similar things, surely those troubles would be liable to be just as marked in the Ubbelohde, and more marked in the Fenske than they were in the U-tube.

He would be very glad if a number of standardizing laboratories would accept one tube in the oil range as being a standard tube. The absolute viscosity corresponding with it might be subject to review occasionally. He would assume that he had a No



2½ tube which gave a time of flow of 200 seconds with a certain oil, and that other standardizing laboratories had a similar tube which would give approximately the same time of flow. Then the instruments used in different commercial laboratories could be compared directly in any country with those few standard tubes. He had always been nervous about standard oils, because he had not information as to how constant they were, but he had never had any indication that properly annealed glass tubes at any time changed their calibration constant.

COL. S. J. M. AULD wrote :—

My feeling for a long time has been that with the viscometers themselves brought to such a pitch of perfection that repeatability is consistently high, the difficulty of reproducibility must be sought elsewhere than in the simple matter of a reading of a time of flow.

While it is true that the baths are laid down to be kept at the correct temperature plus or minus 0.025° F., or a total variation of 0.05° F., I am open to doubt whether this is in practice really accomplished even in laboratories which are well fitted out. Apart from the delicacy of the thermostatic control, which itself must be good in order to achieve the accuracy demanded, I am inclined not to believe that different parts of the same bath have the same temperature. These effects translated into terms of other people's size and shape of bath, thermometers, thermostats, etc., may make thermometric measurements much more diverse than is generally considered.

Even if the difference is only a total of 0.1° C., I make out the effect can be quite marked even on an oil of only average Viscosity Index. On one of the following characteristics :

Viscosity at 210° F.	. . .	20.4 centistokes
" " 100° F.	. . .	368 " "
V.I. " "	. . .	65 " "

I have had some approximate calculations made. As a general rule viscosity variation per 1° C. between, say, 35° and 40° C. is from 5 to 6 per cent., according as the V.I. moves from 100 to 0. An average figure for an oil of the type suggested would be, say, 0.6 per cent. per 1° C., making a difference of 1 unit in the V.I. This difference would, of course, be increased as the viscosity decreases, and could reach a maximum of around 6. These rough calculations would appear to confirm the findings in Table I of the paper.

In connection with the diversity observed between different laboratories, I think one of our first efforts should be that of finding out what is the accuracy of the temperature measurement and control. It would look to me that the differences might easily cover quite a wide range of observed effects.

The following written communication was sent by Mr. ARTHUR W. COX :—

The paper revives once more the thought that we are reaching a time when we need to consider whether the viscosity question of such heterogeneous liquids as petroleum lubricants can be dealt with adequately by one standard practice. There are two aspects of the subject.

The first is that of research. We must continue to investigate the properties of the materials with which we deal, in order to secure the most efficient and economical methods of use. The work must be of a high degree of accuracy, and requires the use of delicate apparatus under stringent conditions of control. It will be time-consuming.

The second use of viscosity measurement is the practical one of scientific control of supplies. It is dependent on the research work just referred to, but does not necessarily require the same type of apparatus or the same niceness of determination or expression. The authors of the paper appear to consider that this practical control work does not require the same conditions as research work, for, on page 3 of the pre-print, when referring to I.P. Method 70/42, they say : " This precision . . . is within the requirements demanded by efficient lubrication." There are others who will agree with this statement.

If, then, it is true that a robust apparatus of simple construction used under standardized conditions can give results which enable one accurately to classify and control lubricants from the point of view of viscosity, there seems to reason no specify delicate and fragile apparatus which requires expensive and complicated controls,



in order to secure a degree of accuracy which is of no practical value. There must arise a time when we must recognize that fine differences in viscosity mean nothing from a practical point of view and that, although our methods may constantly be improved in order to obtain extreme accuracy, the value of the results may be only academic.

A further important point emerges from the paper. It appears that the accuracy of the more scientifically controlled methods may not be so great as the specifications of tolerances would indicate. If this is true there is very good reason for the suggestion of caution in use and interpretation of such results and in the plea for further investigation and consideration. The authors appear to have indicated a weakness in our proposed methods, and I suggest that the problem will be simplified if we remember that we are called upon to consider this question as part of the science of lubrication rather than the science of viscometry.

MR. J. C. CRAGG, in replying to the discussion, said that previous speakers had answered most of the points that had been raised.

The Chairman had said that the new I.P. standard method did not really say that the apparatus would measure viscosity with a maximum error of  $\pm 0.3$  per cent. but that another possible error of  $\pm 0.3$  per cent. must be expected. He thought that this ought to be made quite clear in the method of test, because anyone reading through the method was justified in thinking that any of the viscometers described would measure viscosity with an error not exceeding  $\pm 0.3$  per cent. if all the refinements of the method were observed, and the viscometers were calibrated against any of the calibration standards allowed.

He could assure Dr. Broom that he did not welcome the adoption of viscosity index as a clause in buying specifications, but, as it now seemed to have become established in some Government specifications, it was very unlikely that it could be dislodged.

With regard to the remarks made by Mr. Romney and some other speakers about probable error, he would like to point out that when the authors referred to probable error they had arrived at this quantity by assessing the maximum error and had then calculated the probable error by means of a formula. He quite appreciated the fact that most of the errors might cancel out, but some unfortunate person might make them all on the same side.

He was very interested in the remarks made by Mr. West. It had been well substantiated in the discussion, he thought, that the calibration standards were not interconsistent. It seemed to him that a high order of repeatability could be achieved if due attention was paid to the purely physical side of viscosity determination, if all the viscometers in use were calibrated against one standard only, and if the viscometers were carefully checked to ensure that their calibration constants were true constants. The general run of B.S.I. viscometers did not appear to give reliable results and very little had been said about the Fenske. The general feeling was, he thought, that suspended-level viscometers were likely to be the most reliable type. It was not possible to obtain agreement between different laboratories when all the calibration standards in question were allowed, unless the various laboratories all decided to use the same standard. A small difference in viscosity might influence the viscosity index considerably. It had been shown that a difference in viscosity of 0.7 to 1 per cent. could be obtained by the use of different calibration standards. It was fairly obvious that this state of affairs could have most unfortunate consequences.

With regard to the storage of secondary oil standards, the difference in viscosity on an oil of moderate viscosity was, he believed, about 0.2 per cent. during a year's storage under ideal conditions. A conventionally refined oil stored under similar conditions might show a variation of anything up to 1 per cent.

With regard to the liquid to use in the viscometer bath at 210° F., he could not suggest anything other than white oil, ethylene glycol, or glycerine and water.

In reply to observations that it would be better to convert kinematic viscosity to Redwood viscosity than to convert Redwood viscosity to kinematic viscosity, he would say that the idea of trying to assess the error arising from the calculation of viscosity index from measurements by the Redwood method was based on the fact that there were so many Redwood viscometers in existence. It was necessary to look at the matter from the practical point of view, but the authors were well aware that it was not a practice to be recommended.

He hoped that the various suggestions for arriving at a common calibration standard



would be pursued, as it was quite clear that until all viscosity measurements were based on one standard there would always be some element of doubt existing as to their reproducibility.

Dr. A. E. DUNSTAN, in proposing a vote of thanks to the authors of the paper, said that he had been concerned with the question of the viscosity of oils for more than forty years, and was immensely interested in its development, the methods of determination and so forth, but he proposed to make his contribution to the discussion in the form of a written communication at a later date.

The vote of thanks was accorded with acclamation, and the meeting then terminated.



## WARTIME CHEMICALS FROM NATURAL GAS.\*

*Address before American Institute of Chemists, Chemists' Club, New York.  
23rd October, 1942.*

By DR. GUSTAV EGLOFF.†

FOR many years natural gas has been used mainly for heating and generation of steam and electrical power. In the last few years, however, the tempo of research and development has increased enormously. A much higher field of utilization for natural gas than as fuel is seen in the conversion of the hydrocarbons into superior aviation gasolines, lubricants, synthetic rubber, explosives, acetylene, anæsthetics, plant-life promoters, plastics, solvents, and many other chemical derivatives. A vast supply of these derivatives is available in natural gas produced in this country.

The processes and methods for such conversion in some instances are already in commercial use in the petroleum-refining and other industries. Other methods have been worked out in the research laboratories, and the processes and yields that can be achieved are known; but it remains necessary for further research to establish the means to reduce costs to the level of commercial practicability. Another wide avenue for future development in the natural gasoline and refining industries is thus foreseen.

Natural gas is consumed at the rate of over 2.6 trillion cu. ft. per year. The U.S.A. has proved reserves of 85 trillion cu. ft. and another 85 trillion as potential reserves. The yearly consumption of natural gas is equivalent in fuel value to 100,000,000 tons of coal or over 500,000,000 barrels of fuel oil.

In the Axis countries methane gas is a widely used substitute motor fuel. As a matter of fact, there are over 107,000 compressed-gas (methane, ethane, propane, butanes) propelled motor vehicles operating in continental Europe, which conserved about 2,300,000 barrels of gasoline for military use. The compressed gas is sold at filling stations in exactly the same way as gasoline in the U.S. The gases are compressed under pressures up to 5000 p.s.i. into steel cylinders attached to the running-board of the car or under the floor of trucks and buses. Italy has a number of natural gaswells, and is operating many buses and trucks and a number of locomotives on methane gas. This looks like a desperate measure, since on the average about 135 lb. of steel are required for each of these high-pressure steel-alloy cylinders in order to withstand the high pressures. The products of fermentation of sewage are also used as a source of methane gas in Germany. Methane from coal gas is used for running motor vehicles of one sort or another in many of the European cities. Many coal-hydrogenation units produce gasoline in addition to methane, ethane, propane, and butanes by the Bergius and Fischer-Tropsch processes. The latter process will be re-

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ferred to later in more detail, because it is a potential source of many products. Buses, trucks, tractors, and power shovels, numbering over 25,000 in the U.S., use compressed propane and butane. One truck company has liquefied butane functioning in a dual rôle as a refrigerant for fruits and meats, and as motor fuel for the truck after the cooling has taken place.

Hydrocarbons present in natural gas are methane, ethane, propane, butanes, pentanes, hexanes, and heptanes, etc. The first four of these are gases, while from pentane on they are liquids useful as gasoline.

The individual hydrocarbons have a variety of uses in a number of industries. Propane has been suggested for use as a fuel for breaking in aviation engines on the "testing block," since it has a high octane value and is readily available. Such a procedure would conserve the 100-octane liquid fuels needed for fighter, bomber, and cargo airplanes in the war effort. Other uses for propane which have gained wide commercial application are as refrigerants and solvents in the refining of lubricating oils. Paraffin wax, asphaltic substances, naphthenic hydrocarbons, and other materials are eliminated from lubricants when propane is used as the solvent. Over 50 per cent. of the world's lubricating oils are improved in quality by the use of propane.

Two hydrocarbons of great utility present in natural gas are *isobutane* and *isopentane*. *iso*Butane is the key hydrocarbon in the production of aviation gasoline by alkylation with olefins. In general, there is not enough *isobutane* available, hence it has been necessary to isomerize the normal butane present in natural gas to the *iso*-compound. A number of commercial installations are in operation and under construction to isomerize normal butane to *isobutane*. Normal and *isopentane* are also present in natural gas, and it is highly desirable to fractionate out the *iso*-compound due to its 91 octane rating in contrast to the normal, which has an octane number of 64. *iso*Pentane is blended with aviation gasoline in percentages ranging from 10 to 20, depending on the other components in the final blend.

Olefinic hydrocarbons not contained in natural gas as such, are important for many reactions, not alone for aviation gasoline, but for synthetic rubber and a host of other products. There are several methods of producing olefins from natural gas: one is by high temperature, and the other by catalysis. The olefinic hydrocarbons which are now in great demand are ethylene, propylene, butylenes, pentylenes, and butadiene. Natural gas, particularly the propane-butene fraction, when subjected to high-temperature cracking, produces ethylene, propylene, butylenes, and butadiene. Normal butane is readily converted into butadiene in one or two stages by catalytic means at high temperature, with yields reported of over 60 per cent.

A branched-chain paraffin, *iso*-octane, is of great importance in the aviation fuel programme. There are two methods whereby this hydrocarbon is derived, one is by polymerization, in which *isobutylene* is polymerized to a dimer in the presence of phosphoric acid as a catalyst. *iso*-Octene is the product, and on the addition of hydrogen, *iso*-octane results. *iso*-Octane has an octane rating of 95-100. Another method of producing aviation gasoline is to polymerize the propylene, butylenes, and pentylenes present in cracked gases to a polymer gasoline under selective conditions



which on hydrogenation yields an aviation gasoline blending product of 90 octane rating.

The polymerization process has been largely replaced by the alkylation process, in which *isobutane* is alkylated by butylenes in the presence of sulphuric or hydrofluoric acid catalysts. The resulting alkylate ranges in octane value from 91 to 96. The simplest of the olefins is ethylene, which is used to alkylate *isobutane* at temperatures in the order of 950° F. and pressures up to 5000 lb. This reaction produces neohexane having an octane rating of 94, an important hydrocarbon for aviation gasoline. In addition to alkylating ethylene and butylenes, propylene and pentylenes are also being alkylated with *isobutane* to form additional quantities of high-quality aviation gasoline. It is proposed in some type of operation to utilize the mixtures of propylene, butylenes, and pentylenes in the alkylation reaction with *isobutane*, using hydrofluoric acid as alkylating catalyst to produce alkylate of 91 octane rating, highly suitable for aviation gasoline blending stock.

An important aviation blending fuel of an entirely different type is cumene, which is produced from the alkylation of propylene and benzene in the presence of solid phosphoric acid. A number of commercial units are now in operation.

Normal hexane and heptane are two hydrocarbons which, from a motor-fuel standpoint, are practically worthless unless their molecular structure is changed. Normal heptane has a zero octane rating and hexane about 25. These straight-chain paraffin hydrocarbons, hexane and heptane, can be changed in configuration and also in their properties to improve their anti-knock properties. Hexane and heptane can also be converted into benzene and toluene—two important hydrocarbons for high explosives. Benzene is of vital importance in the production of styrene, which is produced by the alkylation of benzene with ethylene forming ethyl benzene. Dehydrogenation of this compound yields styrene. When styrene (25 per cent.) and butadiene (75 per cent.) are mixed in the presence of a catalyst such as peroxide, polymerization takes place to form the synthetic rubber Buna-S.

The synthetic rubber programme in the U.S. calls for 886,000 tons as planned in September 1942, distributed as follows :

	Types.	Tons.
Buna-S . . . . .		705,000
Butyl . . . . .		132,000
Neoprene . . . . .		49,000

However, the recommendations of the Baruch Committee called for an increase of 220,000 tons to be apportioned as :

	Types.	Tons.
Buna-S . . . . .		140,000
Neoprene . . . . .		20,000
Thiokol . . . . .		60,000

Of the total 886,000 tons of synthetic rubber planned for the U.S., 705,000 tons will come from butadiene and styrene. The normal butane that will be used for butadiene will represent about 80,000 tons a year,



largely derived from natural gas. One plant has a rated capacity of 66,000 tons, and the other 15,000 tons a year.

More than 100,000 barrels a day of normal butane are available from natural gas. If this were used just for butadiene-making, it would satisfy the entire butadiene requirements in our present synthetic rubber programme. Hence it can be seen that the natural-gas industry has more than enough of the hydrocarbons which can be converted into raw materials to supply the entire synthetic rubber programme. Another important source of butadiene is through the dehydrogenation of butylene derived from catalytic cracking. This source will yield at the rate of 283,000 tons annually.

Another type of synthetic rubber is called Thiokol, made by chlorination of ethylene, which is then refluxed with sodium polysulphide. The synthetic rubber programme calls for 60,000 tons of Thiokol yearly. Hydrogen sulphide is present in varying percentages in natural gas, and is readily converted into flowers of sulphur. This sulphur may react with caustic soda to produce sodium polysulphide. After reacting ethylene with chlorine, the dichlorethane is formed which reacts with the polysulphide, yielding Thiokol. The plans call for the use of Thiokol in re-treading of tyres.

Besides butadiene, styrene, and ethylene, a hydrocarbon of great importance is acetylene, used in the production of the synthetic rubber, neoprene; it is also the base material for nylon, a replacement product for silk. Calcium carbide is produced from coal and lime in electric furnaces requiring much electrical power. When water is added to this compound, acetylene is formed.

Natural gas, or products therefrom, under high-temperature conditions readily yield acetylene. Two commercial acetylene units are being installed at the present time, one of which will produce at the rate of 75 tons per day or 27,000 tons a year. Other units are also under way. The charging stock may be either propane, butane, natural gasoline, or fractions from petroleum. It is believed that acetylene can be produced at a lower cost from processing natural gas than by the electrochemical method of producing calcium carbide. The natural-gas industry has enormous volumes of propane and other hydrocarbons available to supply the whole needs of the United Nations for acetylene and its derivatives.

Germany uses acetylene made from calcium carbide, converting it into butadiene by a four-stage chemical process. Neoprene rubber is made from acetylene treated with hydrochloric acid. This type of rubber will be produced at the rate of 49,000 tons a year in the U.S.

The Russians start primarily with ethyl alcohol from grain to produce butadiene. Butadiene from ethyl alcohol derived from grain will be one of the sources in the U.S. synthetic rubber programme. The tonnage of butadiene from grain alcohol will be at the rate of 242,000 a year, representing about one-third of the Buna-S type production.

There are several other types of rubber which are in commercial production, one of which is the type based on *isobutylene* from dehydrogenation of *isobutane* from natural gas or from cracking of oil. *isoButylene* is copolymerized with about 2 per cent. of butadiene or isoprene, resulting in a product called butyl rubber. In the Government programme, this



type of rubber will be produced at the rate of 132,000 tons a year. Butyl rubber, as of to-day, is not as good a synthetic rubber for tyre use as the Buna-S. Tyres made of butyl rubber have a mileage life of about 12,000 miles, with a maximum road speed of 35 miles an hour. This will naturally be improved upon.

There is an important use for synthetic rubber which is fabricated so that billions of minute air-cells are present; it is used as a liner in gasoline tanks in fighting, bombing, and pursuit planes. When a bullet goes through the tank, the rubber causes it to be self-sealing.

Another type of synthetic rubber is the acrylonitrile-butadiene which can also be produced from natural gas, and is known as Buna-N or Perbunan.

One of the necessary materials in the compounding of either natural or synthetic rubber is carbon black, which is made from natural gas. Carbon black is an important component in tyres or other rubber goods, whether it is blended with natural or synthetic rubber, as the properties of rubber, particularly from the standpoint of the tensile strength and wearing qualities under service conditions, are greatly improved. During the year 1940 about 369 billion cubic feet of natural gas were converted, largely by the channel process (air oxidation), into carbon black, with an average of 1.54 lb. per cubic foot of gas or about 285,000 tons, of which about 85 per cent. was used in tyres. Furnace black appears to be superior for use in synthetic rubber.

The world's natural-rubber production for 1941 was about 1,675,000 long tons, of which the U.S. imported over 800,000 long tons. The United Nations have lost over 95 per cent. of the world's natural-rubber sources, and Russia has lost two of its synthetic rubber plants in the Ukraine.

With the tremendously increased demand for airplanes, tanks, motor-trucks, ships, trains, gun mountings, etc., rubber is required in ever-increasing quantities by both the fighting forces and the necessary civilian users for the successful conclusion of World War II. A medium-size tank requires 500 lb. of rubber, small pontoon bridges 1000 lb., for Flying Fortresses, the gasoline tank alone requires 500 lb. of bullet-sealing rubber, while a large bomber uses 1250 lb., gas-masks 0.75 lb., and battleships between 75,000 and 150,000 lb. Tyres for large excavation trucks used by the Army have a diameter of 9.5 feet and weigh over 3500 lbs. There are many hundred more products requiring rubber that are vital in the war effort, such as blimps, barrage balloons, rubber boats, rafts, and life-saving jackets and suits for flyers, hospital rubber needs, etc. Millions of soldiers on the fighting fronts require rubber in one form or another.

The synthetic-rubber picture in the U.S., with its 886,000 tons a year at plant costs of about \$800,000,000, is already well under way. The Baruch Committee has recommended that this tonnage be increased to 1,106,000 tons a year. In addition to being the arsenal for many other war products, synthetic rubber will also have to come from the United States to supply our allies' needs. It may well be assumed that even 1,106,000 tons of synthetic rubber will be too low for the United Nations' requirements.

The question arises now: Is the synthetic-rubber product equal to the natural? In general, one can say that synthetic rubber is at least equivalent to the natural; the chemist's goal is not necessarily to synthesize a duplicate of natural rubber, but it is certain that whatever properties



rubber has that are needed will not only be duplicated, but radically improved and new ones added. Synthetic rubber is superior to natural rubber in gasoline, oil, and chemical resistance. The synthetic product is more stable to light and air, and has greater wearing properties. Some trucks using synthetic-rubber tyres have gone over 35,000 miles. Side-wall tyre strength is greater, meaning greater safety and better road grip. The latter property has been tested out thoroughly on wet and muddy roads. Tests on hills with different trucks have shown that the synthetic-rubber-tyred vehicle goes up a hill with very little side-slipping, whereas the tyres of natural rubber slipped all over the road. On curves, when operating the car at high speeds, the synthetic tyre is safer than the natural.

With the number of research men in the field of synthetic rubber, with expenditure of millions of dollars yearly, one can feel confident that synthetic-rubber tyres will evolve with a life of *at least* 100,000 miles or, expressed in another way, the tyres may well outlive the motor-car.

As far as the author is concerned, synthetic rubber in the U.S. is here to stay, and will be a permanent industry during the next peace period. With the fall of Singapore our greatest source of natural rubber was cut off. Natural rubber (December 1941) cost about 22 cents per lb. The price ranged through the years from 3-5 cents to over \$1 per lb. It can be stated that synthetic rubber in mass production will cost less than 15 cents a lb.

Does this mean that natural rubber will not have its uses? As far as tyres are concerned, it will not have the dominating position it has held heretofore. One may be certain that, good as synthetic rubber is to-day, it will be far surpassed by that yet to come. The properties of the rubber desired will be under close chemical control, based on the high purity of the components started with, catalyst, and precise conditions to yield the finished product. Natural rubbers vary widely in properties, due to many factors. Producers of natural rubber depend on the life-cycle of the rubber tree, climatic and soil conditions, while the chemically produced rubber will have the exact properties for which its structure and use were designed. Natural rubber contains a number of unknown components, whereas synthetic rubber has one, two, or three components of known characteristics.

Natural gas is an important source of high explosives. In some parts of the country it is being cracked into hydrogen, which is combined with the nitrogen from the air, producing synthetic ammonia. The ammonia is readily oxidized with air into nitric acid. Combination of the ammonia and nitric acid produces ammonium nitrate.

In World War I the maximum toluene production was at the rate of 15,000,000 gallons a year, and practically all came from coal-carbonization plants to derive coke for steel-making, with toluene as a by-product. The toluene production in World War II from coal carbonization is at the rate of over 25,000,000 gallons a year. According to published reports, the demand for toluene is from 250 to 300,000,000 gallons a year—the difference between the volume of toluene from coal and the total demand will come from petroleum, *i.e.*, ten to twelve times as much from petroleum. In comparing the two wars, the demand for toluene has increased from sixteen to twenty times. On a T.N.T. basis World War I called for 150,000,000 lb., whereas the *present war calls for 3,000,000,000 lb. a year.* Benzene is readily converted into carbolic acid or phenol through chlorina-



tion and hydrolysis. Combine the phenol with nitric acid, and picric acid, a high explosive, is the result; and when synthetic ammonia reacts with picric acid, ammonium picrate, another high-grade explosive, is formed. The ammonium picrate is relatively stable, but when picric acid is used, it reacts readily with iron to form iron picrate, a very unstable compound that has a habit of exploding when least expected. That is what happened in a number of plants in World War I.

Many natural gases contain hydrogen sulphide, which, when oxidized with air, is converted into sulphuric acid, necessary in so many arts, particularly high explosives. A number of commercial units are producing sulphuric acid based on hydrogen sulphide or elemental sulphur produced from natural gas as a starting material. So we have sulphuric and nitric acid, both produced from natural gases, raw materials necessary for high explosives needed in this present war, some of which are on a scale over twenty times that of World War I.

An important substance in war is glycerine for the production of trinitroglycerine, the commonest form of which is dynamite. The main source of glycerine has been in the splitting of fats to glycerine and fatty acids in the soap-making industry. As is well known, a campaign has been launched by our Government for the conservation of cooking fats of all kinds. These fats have many uses, one of them being the manufacture of soap and glycerine. A few years ago a glycerine-making process was developed by the petroleum industry, starting with propane or propylene, which are chlorinated and then hydrolyzed to glycerine. The glycerine is then nitrated to trinitroglycerine.

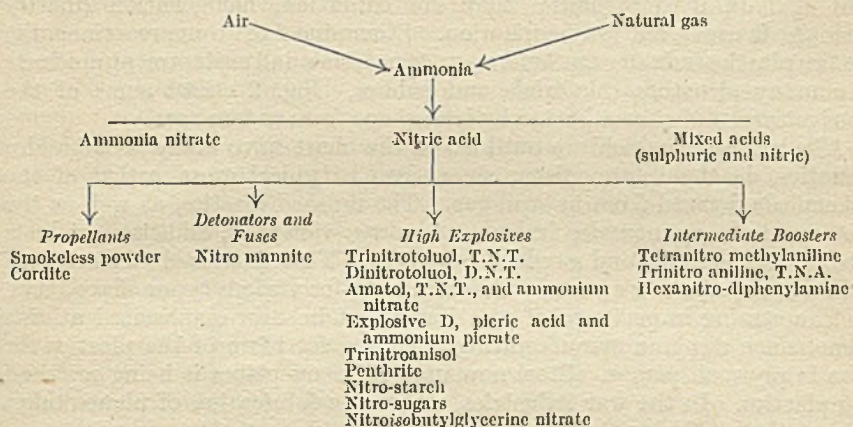
Nitroparaffins from natural gas may well develop into one of the newer and valuable sources of high explosives. Methane gas, when nitrated, produces tetranitromethane. This compound is the most destructive explosive known to man. This product is a very high explosive, extremely difficult to handle. About twenty years ago in a university laboratory in Switzerland, they were working with tetranitromethane which is usually a reaction product of toluene nitration in small concentrations. *Ten grams of tetranitromethane* killed ten individuals, wounded twenty, and nearly wrecked the building. Unfortunately, no commercial process has been worked out to make this product available for use by our armed forces. Fig. 1 shows the importance of nitro-compounds in the manufacture of explosives.

Anæsthetics are vital in a world at war. One of the primary needs of the medical profession has been anæsthetics which have no post-operative dangers due to pneumonia and nausea. Ethylene has been shown to have properties superior to those of ether and nitrous oxide. Deep surgical anæsthesia is readily induced by ethylene, and insensibility to pain comes rather quickly. A general feeling of well-being and comfort, with no harmful after-effects, is present when ethylene is used. *cyclo*Propane, although known since the year 1882, was still a laboratory curiosity until the end of 1930, when it was first applied in human anæsthesia. It has since been rather widely adopted as being one of the safest anæsthetics. *cyclo*Propane, produced from either propane or propylene by a series of chemical reactions, is less explosive in the hospital than either ether or ethylene.



Olefins have been investigated as fruit-ripening agents and for use in inducing an accelerated plant-growth. The first gas to be utilised for this purpose was ethylene, which was first used in an impure form—the fumes from a kerosine stove were found to be effective in bringing about the ripening of citrus fruits. Ethylene was the agent responsible for the ripening.

Nitro Compounds <sup>1</sup>



<sup>1</sup> F. H. Dotterweich, *Refiner*, 1942, 21, #3, 51-6.

FIG. 1.

Ethylene and propylene stimulate potato growth, and have been found to shorten the growth season for tomatoes, grapefruit, and oranges. The German Botanical Society reports that up to a 100 per cent. increase in the yield of potatoes can be obtained when the seeds are treated with ethylene gas. "The tubers of plants grown from seed potatoes which had been exposed for 24 hours to atmospheres containing 7.5, 15, or 30 per cent. gas at 65° F. were more numerous, larger, and richer in vitamin C. It is claimed that this process is cheaper than the application of hormones."

Researches by the Russians on butylene gas showed a stimulating effect on the maturing time of such trees as the walnut, peach, apple, cherry, plum, apricot, and pear. In periods when the season is too short to allow the full maturing, due to winter weather being so prolonged that flower-formation and fruit-setting are delayed, butylene may be used to hasten the growth period. The procedure adapted to plant treatment consists of enclosing the trees in tents or gas-tight covers for about two weeks before the normal or desired leafing—*i.e.*, start of the growth cycle. Butylene is passed into the tent in about 1 part per 100,000 parts of air, at temperatures between 69° and 100° F. for a period of 1-2 hours.

Another method for ripening fruits is to use pentylenes, hexylenes, and heptylenes from cracked gasoline. Addition products of the unsaturated hydrocarbons derived from cracking, such as ethylene di-iodide, ethylene butyrolhydrin, ethylene acetohydrin, and ethylene formohydrin, are also used as agents for furthering the ripening of fruit.

The skin of the green fruit absorbs the olefin or olefin derivative, destroy-



ing the chlorophyll. The advantage in using liquids as ripening agents is that they are absorbed and continue their action after removing the fruit from the liquid; after storage under normal conditions, the ripening is complete.

Natural-gas hydrocarbons can be used as basic materials for the synthesis of plastics after forming olefinic hydrocarbons. There are three basic reactions which in various combinations are used to produce plastics from the derived hydrocarbons; they are oxidation, halogenation (mostly chlorine is used), and polymerization. Stemming from these reactions, the entire plastics industry has built up new materials and replacement products for many structural materials and rubber. Fig. 2 shows some of the derivatives of the unsaturated hydrocarbons from natural gas.<sup>1</sup>

The materials which are outlined in the chart have many uses besides plastics, as the intermediates shown are a typical cross-section of the chemicals derived from natural gas. The finished plastics, as well as the intermediate compounds, find use as paints, varnishes, addition products for lubricating oils, and moulded materials. Their intermediates are used as solvents, cleaning agents, and as additives for various types of products.

The coming importance of plastics cannot be over-emphasized at this time, since airplane manufacturing is calling for more of the plastics for use in plywood planes. Steel now used in many places is being replaced by plastics. In the war industries, where the fabrication of planes takes place, the methylmethacrylate resins are used as the transparent, non-shatterable parts where great visibility is necessary, and this will undoubtedly be used by the automobile industry after the war. A high degree of visibility from all over the motor-car will be worked into the new design, requiring less supporting framework, and a clear-vision rooftop with sliding window will undoubtedly take the place of present designs. In fact, the motor-car of the future may well be substantially all plastics.

At the present time any of the components necessary for the manufacture of plastics of all types can be made from natural gas. One of the important substances for the Bakelite type of plastic manufacture is formaldehyde, made largely from the methanol or wood alcohol by the catalytic reaction of carbon monoxide and hydrogen at high pressures. For a number of years formaldehyde and methanol have been produced from the oxidation by air of natural gas. This process can be readily expanded to produce all the formaldehyde necessary in the ever-expanding Bakelite programme. The phenol or carboic acid and cresols, etc., are also potentially available from natural gasoline.

One big field of research and development that merits discussion is carbon monoxide and hydrogen, or the water-gas reaction to form hydrocarbons. In the U.S. we have over 2.6 trillion cubic feet of natural gas yearly production. This gas contains about 80 per cent. of methane, which can be converted into carbon monoxide and hydrogen by high temperatures in the presence of steam. In Germany the Fischer-Tropsch process has been developed to produce oil from carbon monoxide and hydrogen at the rate of about 15,000,000 barrels a year. The hydrocarbons produced are methane, ethane, ethylene, propane, propylene, butanes, butylenes,

<sup>1</sup> R. L. Wakeman, *Nat. Petrol. News*, 23rd July, 1941, p. R-226.



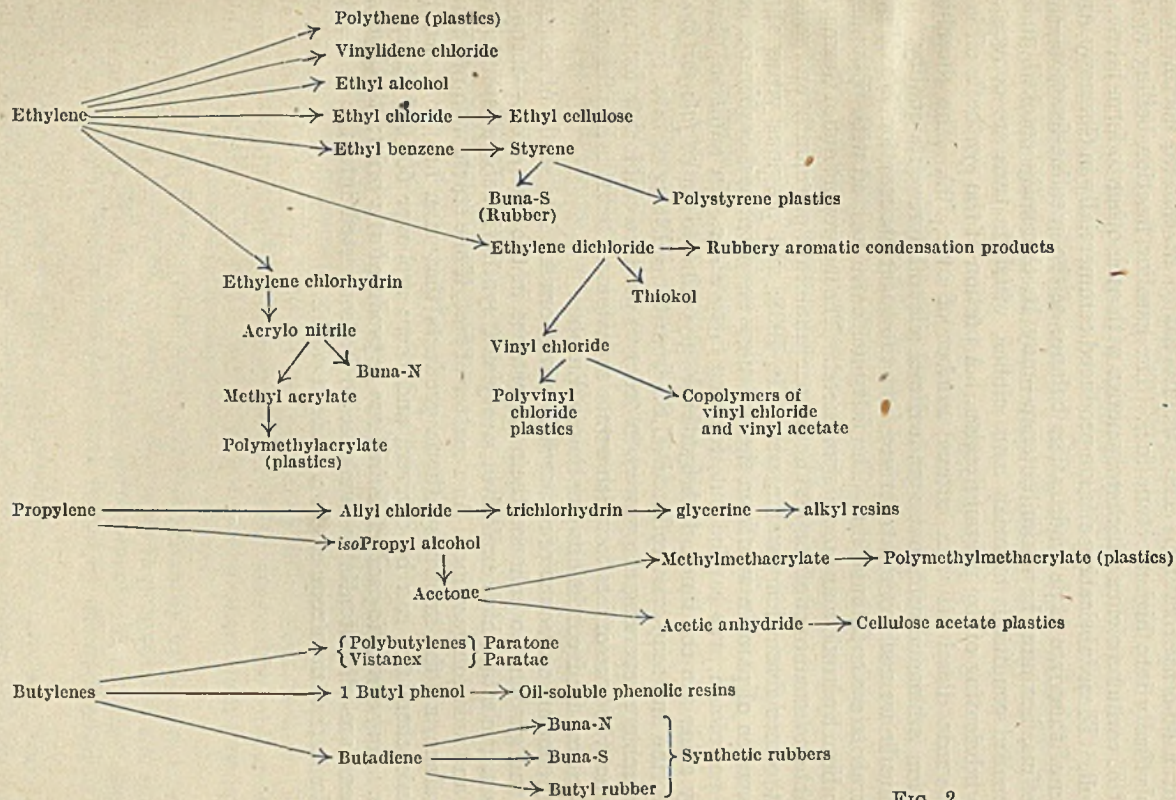


FIG. 2.



gasoline, gas oil, and diesel oil to solid paraffin wax. The reaction takes place in the presence of a catalyst, which may be oxides of nickel, chromium, or cobalt, using temperatures of 400° F. and pressures of the order of 200 lb.

The gasoline produced by the water-gas reaction is poor in quality with about a 40-octane rating. The gasoline has to be cracked thermally or catalytically into higher-octane fuel. The gasoline fraction boiling up to 300° F. contains olefins which polymerize with each other to form lubricating oil. These lubricating oils are produced commercially in Germany, and some of them are high grade. The balance of the gasoline fraction—the paraffins, hexanes, heptanes, octanes—may be thermally cracked under controlled conditions to make more olefins, which in turn are converted into lubricating oil by polymerization. A portion of the synthetic oil is a high-grade diesel oil with octane number of over 100. The diesel-oil fraction is blended with lower-grade diesel oils to improve its quality.

Paraffin wax, which is also derived from the water-gas reaction, is oxidized with air to make fatty acids. The fatty acids are reacted with potassium or sodium hydroxide, and soap is produced. The last report out of Germany is that one small cake of soap is allotted per inhabitant per month, and much of it comes from this paraffin wax. In 1938, and previous years also, conversion of paraffin wax from the water-gas reaction, coal carbonization, and petroleum, was carried out, forming fatty acids. In addition, these fatty acids are combined with synthetic glycerine to make *fats for food*. Glycerine and soap are produced (U.S.) from the splitting of fats, but the Germans are reversing the process in order to produce edible fats for food. It is not the type of fats to which we are accustomed, but it is helpful under the critical food conditions existing in Europe.

An enormous amount of research is going forward in a study of natural gas and gasoline to enhance their importance in the war effort—through solvents, plastics, high explosives, acetylene, synthetic rubber, lubricating oils, and aviation gasoline. As a matter of fact, if one starts with methane gas alone, all the known synthetic products that man has produced in organic chemistry can be derived, and there are over 500,000 different ones. *Any synthetic product desired can be produced at a price*; the hydrocarbons are all potentially available to be converted into the manifold products that man requires in a modern world.



## ABSTRACTS.

	PAGE		PAGE
Geology and Development ...	115 A	Chemistry and Physics of Petroleum ...	140 A
Geophysics ...	119 A	Analysis and Testing ...	142 A
Drilling ...	120 A	Motor Fuels ...	142 A
Production ...	126 A	Lubricants and Lubrication ...	144 A
Transport and Storage ...	136 A	Asphalt and Bitumen ...	145 A
Cracking ...	137 A	Special Products ...	146 A
Hydrogenation ...	138 A	Detonation and Engines ...	147 A
Polymerization and Alkylation ...	138 A		
Refining and Refinery Plant ...	138 A		

### 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.**

Albright, J. C., 351	Degnen, W. J., 381	Langhaar, H. L., 359	Rochner, T. G., 392
Alcorn, I. W., 368	Dennison, E. S., 360	Layng, E. T., 383	Rosen, R., 392
Alexander, W. A., 368	Dodd, C. G., 367	Legge, D. A., 390	
Anderton, B. A., 393	Drennan, H. E., 390	Liberthson, L., 388, 397	Shaw, S. F., 361, 378
Anglo-Iranian Oil Co., 383, 390, 397	Dryer, C. G., 390	Lincoln, B. H., 392	Shoemaker, B. H., 392
Ashworth, D. I., 384		Logan, J. L., 340	Silverman, I., 392
Auld, S. J. M., 392	Egloff, G., 383	Lovell, L. L., 384	Simons, H. F., 357, 370
Ayars, R. N., 337	Ellis, E. G., 392		Simpson, W. K., 390
		Malson, P. E., 384	Smith, E. G., 374
Banks, G. B., 398	Farrell, J. L., 390	Marks, E. M., 397	Smith, S. S., 379
Barnard, E. R., 392	Ferguson, F. A., 384	Miller, W. E., 386	Sterrett, E., 346
Barrer, R. M., 388	Fife, J. G., 397	Mitchell, R. W., 353	Stothart, R. A., 345
Bartell, F. E., 367		Montgomery, J. V., 384	Sudholz, L. H., 392
Barth, E. J., 392	Geddes, B. W., 392	Morway, A. J., 392	
Benner, F. C., 367	Gleason, A. H., 392	Munday, J. C., 390	Thomas, W. D., Jr., 392
Betts, R. L., 384	Grandone, P., 358	Muskat, M., 372	Towle, B. L., 350
Blair, C. M., 388	de Groot, M., 388	Musselman, J. M., 392	Transvaal Chamber of Mines, 391
Blumer, M., 397		McConnell, E. B., 392	Turkevich, J., 382
Bouillion, L. F., 384	Harper, S. H., 395	McKeithan, D. R., 366	Tyson, C. W., 384
Burk, R. E., 384	Henry, R. W., 384	McMillan, F. M., 390	
Bush, R. D., 336	Hilton, P. E., 356		Noel, H. M., 393
Byrkit, G. D., 392	Hirsch, J. H., 388	Oetjen, R. A., 389	Ortynsky, R. L., 393
	Horne, J. W., 385		
Cabeen, W. R., 342	Hunter, A. L., 342	Plaza, J. B., 350	Power, H. H., 350
Chaney, P. E., 352			
Chenick, J. A., 390	Jones, G. W., 386	Randall, H. M., 389	Reed, P., 377
Christianson, L. F., 385	Kao, C. L., 389	Reed, W. W., 384	Rice, F. O., 397
Cook, A. B., 358	Kasline, F. E., 335	River, W., 384	
Cook, E. W., 392	Katz, D. L., 376		
Cozzens, F. R., 369, 373	Kearby, K. K., 381		
Craft, B. C., 375	Keiser, B., 388		
Crown, W. J., 338	Keith, A., 341		
	Kennedy, R. E., 386		
Dahlgren, E. G., 365			

### Geology and Development.

**335. Edison Oil-Field.** F. E. Kasline. *California Oil Fields*, Vol. 26, July 1940-June 1941.—The Edison oil-field is situated about 9 miles south-east of Bakersfield, Kern County, and has a proved acreage of 1360 acres, divided between North-west Edison area (110 acres), West Edison area (120 acres), and Edison area (1130 acres). According to this report, the first completion for the entire area was in the North-west Edison district in 1928, and up to the end of 1941 there were ten producing wells in the same area, two of which were idle. Production is from the "Wicker" sand, a sandy interval found at the base of the Fruitvale shale. The area is complicated by faulting.

In 1931 two wells were drilled in Edison area and 50 bbl. per day of 13.7° gravity oil were obtained from one of them. No further drilling was done until 1933, when Monterey Exploration Company acquired certain rights. Drilling continued, and by



the end of 1934 there were ten producing wells in the area. In 1937 and 1938 five wells were completed and seven unsuccessful ones were abandoned. In 1939 and 1940 there was no new activity, but in 1941 eight new wells were completed.

West Edison area was first proved in 1935, and in January 1939 nine wells were being pumped at the daily average rate of 55 brl. of oil and 8 brl. of water. In 1940 four new wells were drilled, and in 1941 five, raising the total number of producing wells to eighteen. Throughout the field, water suitable for irrigation may be obtained from sands at depths between 200 and 600 ft.

H. B. M.

**336. Résumé of Oil-Field Operations in 1940.** R. D. Bush. *California Oil Fields*, Vol. 26, July 1940-June 1941.—It is reported that total production in the State of California for the last six months of 1940 amounted to 112,323,908 brl. of oil and 108,615,985 brl. of water. The total for the whole year was thus 223,292,063 brl. of oil, a decrease of 961,730 brl. as compared with 1939. Nevertheless there was a substantial increase in production of oil in the second half as compared with the first half of 1940.

Tables included in the report give summarized production records of the five State districts over six-month periods of 1939 and 1940; segregated data of production of clean oil and water in various fields for the half-year ended 31st December, 1940; average daily and closed-in production by months for each field for the year 1940; and average daily production and disposition of natural gas by fields monthly for the year 1940.

At the end of 1940, as computed from American Petroleum Institute statistics, there were 145,566,524 brl. of crude and refined petroleum in storage in Pacific Coast territory. This represented a decrease of 6,368,724 brl. compared with a decrease of 5,749,041 brl. in 1939.

6,467,000 brl. of crude and refined oil were shipped to Atlantic Coast ports during the year, or 491,000 brl. less than in 1939.

On 1st February, 1940, prices of crude oil were decreased: heavy crude oils were cut 15% while higher-gravity oils were cut proportionately less.

During 1940 only one new oil-field, Del Valle in Los Angeles County, was discovered, but new productive areas were developed in already known fields, chiefly at Dominguez, Rosecrans, and Wilkington in Los Angeles County and at Trico Gas, Kern County.

H. B. M.

**337. Webster Area of Midway-Sunset Oil-Field.** R. N. Ayars. *California Oil Fields*, Vol. 26, July 1940-June 1941.—The Webster area of the Sunset oil-field of the San Joaquin Valley is situated in low foothills cut by numerous erosional valleys. In 1920 R. W. Pack (U.S.G.S. Prof. Paper No. 116), described two producing zones in the area, but since that time a deeper Miocene producing interval has been located, known as the "Webster" zone. This report describes the deeper strata and correlates Pack's findings with the further information now available.

Structurally, the Webster zone interval is the upper end of a plunging anticline which ends in an elongated dome where the anticline intersects the monoclinical trend of the beds along the west side of the valley. There is approximately 400 ft. of closure on the dome. The zone is from 1600 to 2100 ft. and has a total thickness of from 200 to 300 ft. In the upper 150 ft. the sand averages about 50% of the total thickness, but in the lower part the proportion is considerably less. In most cases the oil-saturated sand is found in the first 200 ft. of the zone. The oil-sands are not continuous, neither do they occur at the same stratigraphical horizon throughout the area owing to the lenticular character of the formations.

Total maximum proved acreage of the Webster zone is reported as 315 acres with potential locations for sixty-three wells with 5-acre spacing; twenty-seven commercial wells have been drilled. Wells below the minus 1000 contour in a heavier drilled area to the south-east make considerable amounts of water, in one case up to 90%. Total initial production from all twenty-seven wells was 2964 brl. of oil, 13.9-15.6° gravity, and 261 brl. of water per day. In September 1940 the total for twenty wells was 813 brl. of oil and 2234 brl. of water per day. These figures indicate the small amount of water produced originally as compared to the large amount now being produced.

H. B. M.



**338. Wilmington Oil-Field.** W. J. Crown. *California Oil Fields*, Vol. 26, July 1940-June 1941.—Wilmington oil-field is situated in the harbour district of the City of Los Angeles and has all the advantages of a field in the immediate vicinity of refineries and a busy harbour. According to this report, it is of especial interest, in that it was the first large town-lot field in which the State law was applied to regulate the spacing of wells; it is one of the few multiple-zone fields discovered in many years; of the Los Angeles Basin fields, it is second largest in areal extent; it contains one of the largest reserves of oil in the State; gasoline obtained from the crude oil has probably the highest octane rating in the entire State; and oil-zones are shallow.

Structurally, the field is classed as an anticlinal fold with its main axis trending in a north-westerly-south-easterly direction and cut by a series of transverse faults which in some cases branch out into minor faults.

Production is obtained from four zones: Tar and Ranger of Pliocene age and Terminal and Ford of Miocene age. Oil in Tar zone, as its name indicates, is heavy, with a gravity range of 13-15° A.P.I. Within the Ranger zone there is a considerable change in sedimentation and the widest gravity range of any zone is found—i.e., 14-24° A.P.I.

The Terminal zone is normal as to sedimentation. In the upper part, gravity of the oil varies between 17° and 26° A.P.I., while in the Lower Terminal zone it ranges between 27° and 31° A.P.I. Production in the Ford zone comes from a 700-ft. interval of which 150 ft. is sand. Gravity of the oil is between 29° and 31°. With the exception of those in the Ford zone, all the sands are relatively high in porosity and permeability.

It is recorded that a large part of the field, chiefly where only the Ranger zone is productive, has not yet been developed, and it is the author's opinion that if and when all zones are developed by opening up undrilled portions, ultimate recovery from all zones will be approximately 700,000,000 brl. By 1st January, 1942, 140,201,679 brl. had been produced, leaving an estimated reserve of 559,898,321 brl. Hitherto, the field has averaged 35,050 brl. per acre, and it has accordingly a potential ultimate recovery figure of 175,000 brl. per acre. It is further estimated that the future life of the field will extend over more than thirty years, and that it will play a major part in the Californian oil industry for many years to come.

H. B. M.

**339.\* War and Wildcatting.** Anon. *Petrol. Engr*, December 1942, 14 (3), 14.—For the past ten or twelve years the U.S.A. petroleum industry has been in a position to produce a raw material and its finished products in quantities far greater than the consumptive demand, but this condition no longer holds. The expanding need for petroleum products will fully test the ability to produce and manufacture, and a maximum rate of withdrawal from underground oil reserves seems imminent. Frequently the U.S. oil reserve figure has been given as 20,000,000,000 brl., and many have accepted this quantity as being available for the war effort, but the quantity which is significant is that which will be available during the next two or three years. This latter is not the same as the figure quoted above.

The decline in discoveries over the past five years, the lack of development in proven and semi-proven areas, and the cessation of wildcatting for new reserves in twenty oil-producing States show that the U.S. production will be approximately 1,349,650,000 brls., 1,283,100,000 brl., 1,189,525,000 brl., 1,086,555,000 brl., and 995,610,000 brl., in 1943, 1944, 1945, 1946, and 1947 respectively.

A study of the history of oil production and the economics of various methods of recovery reveals that it would take forty years to recover 16,360,000,000 brl. of the estimated reserve of 20,000,000,000 brl. In the period 1943-7 inclusive only 5,904,000,000 brl. are likely to be available without severe water contamination.

The barriers to wildcatting must be lowered, and the wildcatter should be given a price which will encourage speculative undertakings, or else there will be a shortage of crude or of the required types of crude.

G. D. H.

**340.\* November Wildcatting Fails to Provide Adequate New Fields.** J. L. Logan. *Oil Wkly*, 21.12.42, 108 (3), 14.—In November the number of exploratory tests completed was only 75% of the average number set by the C.P.O. as the monthly goal this year. Few if any major strikes were made, and two-thirds of the new fields and pays were in areas where the discoveries are generally not large—namely, Illinois, Indiana, Kansas, North Texas, and West Central Texas. In spite of con-



siderable wildcatting, no discoveries were made in California, Arkansas, Mississippi, New Mexico, East Texas, Upper Texas Gulf Coast, Louisiana Gulf Coast, Michigan, and Kentucky.

Eighteen new fields and eleven new pays were discovered, a total of only twenty-nine instead of the monthly average of fifty-two hoped for. Eight gas-fields were discovered, while ten were expected. The Robertson field of West Texas is one of the more important November finds, and produces from the Permian at 5010-5950 ft., but more development is needed to fix its value. It is on an anticline located by geophysics and subsurface geology. Oil was also found in the Clear Fork sandy lime of the Monahans field, the new pay coming in at 759 brl./day from 5633-5643 ft. The previous development was in the Ellenburger at 10,345 ft. A deep high-gravity oil-field and new pay, and a shallow oil-field were found in Jackson County on the Lower Gulf Coast of Texas. In Young County two new Mississippian pays were opened, and North Texas had two other new pays and three new fields. A shallow gas-field and a minor shallow oil-field have been found in West Central Texas. Ten-sleep oil production has been obtained in the Elk Basin field of Wyoming, which has given oil from the Frontier sands. The new Horse Creek field of Laramie County, Wyoming, yields oil from the Lakota at 5451-5510 ft.

Tables give the results of wildcatting in November 1942, by States and districts, and list the new oil-fields and pays, with data on depths, formation, production, type of structure, date, and method of discovery, etc. G. D. H.

**341.\* Are Salt Domes Genetically Related to Zones of Crustal Megashearing?** A. Keith, *Oil Wkly*, 21.12.42, 108 (3), 24.—The principal zones of megashearing of the Gulf Coast area have been mapped, primarily on in-line evidences of vulcanism in its many forms, and on the bulky data about the co-axiality of many recognized structures, with extrapolation across the numerous intervening masked areas. It has been noted that nearly all the more important salt domes of Louisiana are within or near the two east-west zones of crustal mobility. These are tangential to the Gulf coasts. In Texas the principal domes are near the great negative arc which defines the coastline, and a rectilinear zone which transects Louisiana from the north-east and is tangent to the Texas coastline near Corpus Christi. Submarine salt-domes have been found bordering a long, deep, nearly rectilinear trough, which is in line with another mega-structure.

Minor alignments of salt-domes have been noted for a long time. Seismological data show that the great zones within which most of the salt-sulphur domes lie have also been centres of numerous seismic disturbances.

It seems that the shaping of the Gulf has been attended not only by strong downward crustal bending, but also by deep near-shore penetration of the crust. This assumption gives a reasonable solution of a number of puzzling salt-dome problems, and suggests the whereabouts of the more promising areas for future drilling. G. D. H.

**342. Geology of North-west Wilmington and Torrance Fields, California.** W. R. Caben and A. L. Hunter. *Oil Gas J.*, 14.1.43, 41 (36), 70.—The Wilmington oil-field is a transversely faulted anticlinal nose, plunging north-west. The various fault blocks differ as regards oil gravity, gas/oil ratio, productivity, and edge-water limits. Shallow production comes from the Tar zone of Middle Repetto (Pliocene) age. The Ranger (Pliocene) and Terminal (Miocene) zones are not productive throughout the town-lot area. The Ranger zone is 240 ft. thick, and lies immediately above an unconformity. The Ford zone is productive only in the south-east part of the town-lot area. It is 700-800 ft. thick. The Lower Terminal and Ford zone possibilities have not been fully exploited.

The Main zone production at Torrance is roughly equivalent in age to the upper Terminal of North-west Wilmington, while the Del Amo producing zone is roughly equivalent to the lower-middle and upper-lower part of the Ford zone at Wilmington. Torrance is a complex structural nose plunging south-east, and generally agreeing with an irregular ridge of the basement schist. The north-westerly rising schist ridge changes direction when it nears the coast and becomes more northerly in trend. The Del Amo production is generally limited to the south-west flank of the structure, while the Main zone production is chiefly on the north-east flank. The structural closure decreases with distance above the basement, but not so rapidly as would be



expected if it were wholly due to thinning and differential compaction of the beds over the ridge. Near the Torrance Harbour City area the Torrance nose dies out in a south-easterly direction into the flank of the Wilmington structure, whereas the Wilmington nose dies out in a north-westerly direction into the flank of the Torrance structure.

The movement of the faults of the North-west Wilmington plunging nose was largely horizontal, little vertical movement being apparent.

G. D. H.

**343.\* December Wildcatting Yields but One First-Class Field.** Anon. *Oil Wkly*, 18.1.43, 108 (7), 18.—During December 1942, 201 wildcat wells were completed, less than two-thirds of the number considered to be the safe minimum monthly average. The discoveries were mainly of a secondary or minor importance, 17.9% of the wildcats being successful. There were nineteen new oil-fields, three distillate fields, four gas-fields, and ten new pays in established fields. The O.P.C. programme is based on the drilling of sixty-two successful wildcat wells per month, ten of which should prove new gas-fields.

Only one out of the thirty-six discoveries seems likely to be an important field, although a second is important because of its remoteness from areas of established production. The important strike was at Quitman in Wood County, East Texas, 7½ ml. south-west of the young and important Coke field. The discovery well flowed at the rate of 726 bbl./day, with a G.O.R. of 316 and a casing pressure of 660 lb./in.<sup>2</sup>. The oil is from the Paluxy at a depth of 6280–6310 ft. The field is on an anticline outlined by geophysics and geology, and it may have further pays in the Lower Trinity.

The East Strand field of Kern County, San Joaquin Valley, discovered late in November 1942, is estimated to have a reserve of at least 5,000,000 bbl. A 125-bbl./day pumper has been opened in Clarke County, Mississippi, and this field promises to lead to more prospecting which may extend into Alabama.

A new shallow field and new zones in three established fields have been opened in Illinois. About 50 bbl./day were obtained from the discovery wells of each of the five new fields of Kansas. Oklahoma had a new oil-field, a new gas-field, and a new pay. A poor producer was completed in Wise County, North Texas.

A table summarizes the results of wildcatting in the various States and districts in December 1942.

G. D. H.

**344.\* New Oil- and Gas-Fields and New Pay Horizons Discovered in the United States.** Anon. *Oil Wkly*, 18.1.43, 108 (7), 19.—A table gives data about the thirty-six successful wildcats completed in December, and in some cases in November 1942. The data include name, county, company, well location, distance from pipe-line, date spudded, date completed, total depth, depth of completion, name, age and type of producing formation, initial production, mode of production, oil gravity, type of structure, and method of discovery.

G. D. H.

## Geophysics.

**345.\* Radioactivity Determinations Set Production Delimitations.** R. A. Stohart. *Oil Wkly*, 4.1.43, 108 (5), 19.—The precise determination of radioactivity at the earth's surface is not now generally considered to be capable of locating possible oil reserves, because of the inability to distinguish between emanations from oil deposits and from certain buried minerals. However, it now seems to be possible clearly to define the productive limits of a field once a well has found oil and the radioactive characteristics of the locality have been determined.

Crude oil has a relatively large capacity for absorbing radioactive emanations, and this feature is apparently its sole source of radioactivity. It absorbs 40–50 times as much radon as does water at temperatures between 20° C. and 50° C., and so only hot, highly mineralized waters are likely to approach crude oil in radon-absorbing capacity. Natural gas does not absorb radon. A variety of rocks which might provide the emanations absorbed by crudes have been examined, and also the crudes from the fields where the rocks were obtained. The crudes were found to contain several times more radon emanation than might be expected from a study of the rocks.



In finding the limits of production in an area with a producing well, 4-ft. holes are drilled, and a sample of gas is drawn from the bottom of the hole for a determination of its degree of radioactivity. On plotting a profile of the intensity of emanation, the best oil production is found in the crestal region of the profile. The method has been tested on wedge, fault, and other types of oil-fields, and good agreement found with the drilling results. The thinning of oil-sands is also indicated.

The method may reduce, if not remove, the need for drilling a series of dry holes in order to define the limits of commercial oil production in a field. G. D. H.

### Drilling.

**346.\* Revised Operating Practices Prolong Rig Service.** E. Sterrett. *Oil Wkly*, 9.11.42, 107 (10), 41.—In a long paper, maintenance hints and methods are detailed with a view to lengthening the life of drilling equipment. Mud pumps and lines, draw-works and their component parts, drill-strings and bits, boilers, and paint on all this equipment are each discussed separately. Since the preservation of the metal is dependent on the continuity of the protective coating, paint now stands high in the list of maintenance "tools," and regular painting is incorporated in the work schedule. Equipment which is temporarily idle, such as sand-line equipment, is cleaned and painted at the first opportunity. Cleaning, not just wiping off with a rag moistened with kerosine, is now required before the paint is applied. Where steam is available on the rig, most parts which have acquired coatings of dirt and grease are steam-cleaned by flushing off all surfaces with a jet of steam, loaded with water or kept as dry as possible, depending on the equipment being cleaned and the arrangements for proper drainage of the run-off.

If the equipment is paraffin-coated, as is often the case on work-over jobs, the addition of some commercial solvent to the steam-jet greatly speeds up the cleaning, and also leaves the surface in condition for painting without the need of wiping down. An item of equipment, steam-cleaned, will dry quickly unless pockets retain condensation, and the painting may follow closely behind the steam hose. Care must be exercised with the use of the steam-jet to prevent the force of the flow forcing grit and water into close clearances in the joints of certain equipment, packing, and similar points; but for the preparation of broad surfaces the jet is fast and saves much hand labour. Flame-cleaning of drilling-rig equipment is confined to relatively few items of the operating class, though it may be used on boilers, stacks, steel mud-pits, flumes, steam- and mud-lines, without injury to the material being prepared for painting. It is also useful in cleaning the racks made of old pipe, but should not be used on any galvanized steel, such as derrick substructure and beams, even though the condition of the galvanized coating is so bad as to require painting as a matter of preservation rather than to preserve appearances. The procedure of painting and maintaining the coating is detailed.

Hot enamel coatings, such as those used for protection of pipe-lines, are being used by some companies for much of the equipment formerly considered fully expendable. The big-inch pipe used as mud flumes on many rigs, for instance, is thoroughly cleaned, and then given a brush coat of enamel inside and out. This coating is highly resistant to the scouring of the cuttings carried in the mud-stream, and if given proper care during location shifts, the flume will outlast several using bare pipe alone. Other items coated with hot enamel are water- and mud-lines, pump-skids, and other equipment which must lie on the ground throughout the drilling programme. The enamel may also be used on steam-lines to advantage if they are to be lagged, but otherwise the temperature of the steam inside the lines is too high to permit the coating to retain the desired hardness. A. H. N.

**347.\* Rebuilding by Metallizing for Surface-Wear Extension.** Anon. *Oil Wkly*, 9.11.42, 107 (10), 64.—Wear of bearings and other contacting parts in oil-field equipment may be segregated into two general classes: that in which the wear impairs the structural strength of the units and increases hazards; and that in which abrasion increases clearances and encourages leaks without reducing the initial factor of safety below the point of safe operation. In the latter class may be set much of the wear on such reciprocating parts as pump plungers, engine pistons, crossheads and guides,



and compressor rods; oscillating parts such as pumping-unit irons, plug-valves, and cam-follower pins; and rotating parts such as cam-shafts, crankshafts, sheaves, and follower rollers. Almost without exception, design of these parts is to a specified factor of safety, with the limiting dimension being taken as the next commercial size greater than that indicated by evaluation of the design formulæ.

Where such wear is known or may be demonstrated to have left unimpaired the initial design factor of safety, application of additional metal through metallizing enables the low spots to be built again to dimensions, the desired surface finish again attained, and the part thus restored to service. Metallizing is not to be considered as a welding operation. No fusion of metal between that of the part under repair and the stream of molten particles issuing from the spray-gun is attained. The bond is purely mechanical, and as such cannot be considered as adding to the tensional, torsional, or bending strength of the part. The sprayed film is, however, resistant to compression or crushing to the extent to which the added metal is itself resistant or, if hard metal is being applied over softer core material, to the extent the core is resistant.

The method is described in detail (*see* Abstract No. 363 in the Production section for similar description).  
A. H. N.

**348.\* Chain-Drive Maintenance Pivots on Lubrication.** Anon. *Oil Wkly*, 9.11.42, 107 (10), 72.—While the installation of a chain is not difficult, reasonable care should be used in its operation and maintenance. The greatest handicap to the dependability and serviceability of roller chains is avoidable abuse. Periodic inspection, good maintenance, and, most of all, correct lubrication, will prevent the necessity for replacing chains and sprockets prematurely. To secure maximum life and efficiency, the three factors of chain alignment, chain adjustment, and chain lubrication should be frequently checked to ensure preservation of conditions attained at time of installation.

Shafts must be parallel, level, and rigidly mounted. Sprockets must be in line with each other and securely fastened to the shafts. Inner faces of inner links should be examined for wear from contact with the teeth of sprockets improperly adjusted for all drive factors. The sides of the sprocket teeth should be examined for wear, this being consequent to misalignment and non-parallelism of shafts, and conditions thus indicated should be corrected before the maladjustment has caused serious deterioration of the drive. Link-plate outer edges should be examined, and any highly localized brightness should be regarded with suspicion as this, in common with tight joints, may be traced to the running of the chain with inadequate clearance, the links striking against some obstruction. Centre distances should be adjusted occasionally, on such drives as permit this aid to prolonging drive life, to take up slack as it develops.

Details are given for the proper use of lubricants and for the correct placing of idlers to ensure maximum life of chains. Chain repair is briefly discussed.  
A. H. N.

**349.\* Better Wire-Rope Care Will Pay Big Dividends.** Anon. *Oil Wkly*, 9.11.42, 107 (10), 104.—Cleaning wire ropes before lubrication is fully discussed. Since not only the individual wires, but the strands themselves tend to open up and move with relation to one another when passing around a sheave, lubricant should be applied at a bend in the line if at all possible. If the sheave itself is so located as to prevent the application at this point of principal bending, the lubricant should be added just before the rope, in normal travel, would pass between the flanges. Also, when running a line for lubrication, the usual high speed should be cut down so as to allow a brief period for the lubricant to penetrate between the wires and find its way to the core. The lubricant should be acid-free and not water soluble. It must form a tenacious film and penetrate readily, as tiny interstices open between wires. If any doubt exists as to the proper lubricant to apply, the specifications available from any wire-rope maker should afford an accurate guide.

One function of wire-rope lubrication, which is little understood by many users, is that of keeping the hemp core soft, pliable, and swelled to proper size. A dry core will shrink, withdraw its support from the strands, and allow them to crush one another, as well as to break down the fibres of the core itself. A well-lubricated core acts as reservoir for lubricant, feeding a little to the adjacent wires at each flexing over a sheave. On the contrary, a core lacking lubricant to fill the pores of the fibre, will



soak up water during wet weather, keeping the inner wires moist and greatly encouraging the formation of rust, with eventual weakening or breaking of the wires thus exposed to severe conditions. Different types of lubricants are discussed with their special uses. Proper methods of spooling and general maintenance of ropes in use are indicated.

Replacement of smooth drums with spirally grooved units adds greatly to the life of a line. There is available a spiral grooving which may be welded or brazed to a smooth drum, and no dismantling or change of equipment be required. Drums which have worn may be rebuilt by metallizing or welding while still in place, and should be checked after repair to ensure the proper groove depth and pitch. If concentric grooves are used, the depth should not exceed 10% of the rope diameter, or severe crushing will be sustained by the rope at each crossover. The spiral groove should be of sufficient depth to support the rope through an arc of contact of 150°, and clearance between adjoining grooves should be such as to insure  $\frac{1}{8}$ -in. space between adjoining turns of the line on the drum for all sizes from  $\frac{1}{2}$  to  $1\frac{1}{2}$  in., and  $\frac{3}{16}$  in. for larger sizes. A drum re-worked to include these factors may cut the loss through drum crushing to one tenth of that formerly sustained. A. H. N.

**350.\* Hydration-Pressure Relations in Clays and Heaving Shale.** H. H. Power, B. L. Towle, and J. B. Plaza. *Oil Gas J.*, 12.11.42, 41 (27), 215. *Paper Presented before American Petroleum Institute.*—Some shales are thought to heave into the bore-hole of drilling wells because of the inclusion of varying quantities of clay materials which swell and expand when wetted by water from the drilling mud. The expanded shale not only fills the hole a large distance above the point of entry, but in some cases also exerts sufficient pressure to crush the steel casing. A survey of the literature indicates that little has been written concerning the swelling of clay minerals. Most of the literature on swelling is confined to descriptions of experiments performed with the so-called elastic gels. Hence, opportunity exists for research in this field, with due regard for the accomplishments to date in allied investigations.

Accordingly, a programme of investigation was initiated in the petroleum engineering laboratories of the University of Texas in order to determine the hydration-pressure relationships existing in clay materials, such as Wyoming bentonite, when wetted with water. The conclusions reached from this work indicate that certain clay materials are capable of producing pressures of considerable magnitude when confined during hydration. The effects of certain chemicals on pressure increases or decreases are also shown. It is also apparent that the nature of the exchangeable bases present may influence the swelling-pressure characteristics of the clay material.

The present work was intended only as an extended test of the hydration-pressure bomb. No single line of investigation has been followed rigidly. It is expected that future work with the bomb will make possible more thorough investigations, particularly from the standpoint of pressure reduction by means of added chemicals. From the data gathered, the following conclusions have been reached: (1) Materials of the type represented by Wyoming bentonite are capable of producing pressures of considerable magnitude when confined during hydration. (2) Although the data on this point are not conclusive, it is thought that the nature of the exchangeable bases present will largely determine the swelling-pressure characteristic of a clay material. (3) A study of existing information regarding the increase of thickness and charge of the outer layer of the micelle as influenced by added electrolytes may provide a means for predicting relative maximum pressures attainable in the apparatus described. Additional experimental work will be of interest concerning such predictions. (4) In the early states of hydration, the swelling process follows a relationship of the type:

$$P = KT^n.$$

A. H. N.

**351.\* Adjustability and Interchangeability Feature Rig Substructure.** J. C. Albright. *Oil Wkly*, 16.11.42, 107 (11), 45.—The substructure is made of casing and line pipe all of three identical sections to facilitate interchangeability. The three identical sections are made with pipe, the vertical sections of which require reasonably large-diameter pipe in pieces about  $4\frac{1}{2}$  ft. long, cut squarely on one end for welding into the channel of the channel iron, which can be any length desired. Diagonal braces are



attached at an angle of  $45^\circ$  to the channel iron and the vertical riser, to obtain the required stiffness to resist vibration and strain of supporting heavy machinery in motion. This 8-in. riser is equipped with a number of  $1\frac{1}{4}$ -in. holes drilled on 5-in. centres squarely through both walls. To provide equal interchangeability, the first riser made was used as a pattern for the remaining units, so that the distance from the channel upwards to the first hole would be exact. The overall length of the riser is not so important as the spacing of the holes, since the upper end comes in contact with no other section, and that end can be left with threads or cut plain. Rough edges are eliminated.

Horizontal members on which the skid-frames of the engines and draw-works are placed, after the substructure is completed at the location, are made of three sections of pipe slightly smaller than the 8-in. vertical risers. Legs welded to the long horizontal supporting members are placed so that the skid-frames of the engines and draw-works rest directly above the vertical supports with the channel-iron footing on timber mats. These legs, one on each end of the horizontal pipe, have drilled holes of the same size as those which are placed in the vertical risers, but on closer centres, so that a relative fine adjustment in height can be made by matching certain holes in the inserted pieces and the vertical risers. Ribs welded to the outer surface of the insert pipe walls have sufficient thickness to provide a close fit between the inserts and the vertical risers, so that sufficient rigidity is obtained to eliminate side sway. Ribs have been found to serve better than a squeeze-fit pipe, since the bearing surfaces are smaller, and when the ribs are greased, inserting and removing the sections should not be difficult. The paper is illustrated.

A. H. N.

**352.\* A Review of Recent Advances in Drilling-Mud Control.** P. E. Chaney. *Oil Wkly*, 23.11.42, 107 (12), 25. *Paper Presented before American Petroleum Institute.*—

A general view of the colloidal chemistry of clays and shales is presented as a theoretical background for the explanation of the various mud problems, and for the chemical treatments used in their correction. Each of the common mud problems is discussed separately, and an attempt is made to provide a method of diagnosing the cause of trouble from changes in the physical properties of the mud. Following this diagnosis, one or more methods of treatment for correction of the trouble are described, and the theoretical explanation for the action of the corrective treatment is presented whenever possible. A table showing the combinations of mud properties, or "symptoms" associated with each type of trouble, together with an outline of the corrective treatment, is presented as an aid in deciding rapidly on the cause of trouble, and in selecting the proper treatment. Two of these are reproduced as illustrations. Rock salt or salt-water flow: symptoms, (1) rapid increase in salt content of mud; (2) first effect is increased gel rate and viscosity; after several hours, or perhaps a day, viscosity drops below normal; (3) water loss increases rapidly; (4) in case of salt-water flow, mud continues to flow after pumps have been shut down. Chemical treatment: (1) In case of salt-water flow, increase mud weight as rapidly as possible until shut off is obtained, but watch closely for loss of mud to the formation, and stop weight increase if this occurs (even though some flow of water continues) until mud loss is stopped by addition of plastering agents. (2) In extreme cases, pull up in casing to condition mud, as the danger of stuck drill-pipe is serious when salt water is encountered. (3) Add large quantities of highly colloidal clay, preferably hydrated in fresh water before addition to the mud. (4) Copious additions of Quebracho are sometimes helpful in reducing "salt-water gel." (5) In severe cases, substitute organic colloids for clay in mud.

Heaving-shale troubles are treated as follows. A. Bentonite heaving shale: symptoms, (1) increase in mud viscosity and gel strength; (2) reduction in water loss and cake thickness; (3) tight places in hole. Chemical treatment: (1) Reduce water loss to lowest possible value. (2) Reduce mud pH to 7.2-7.6 by use of pyrophosphate. (3) Low pH muds tend to have very low gel strength. Do not reduce pH low enough to prevent suspension of weighting material and cuttings. (4) Keep mud viscosity and gel strength as low as possible to prevent swabbing. (5) Use sodium-silicate mud in very severe cases. B. Gas-bearing heaving shale: symptoms, (1) sour-gas odour at shale shaker; (2) little increase in viscosity unless bentonitic shale is also present; (3) tight places in hole. Chemical treatment: (1) Reduce water loss of mud. (2) Increase mud weight until difficulty is stopped or until loss of mud to forma-



tion begins. C. Plastic flow heaving shale: symptoms, (1) large slabs of shale circulated out of hole; (2) shale may flow into hole to a height of a hundred or more feet on removal of drill-pipe; (3) stuck drill-pipe or tight places in hole; (4) usually found on highest part of structure. Chemical treatment: Same as for "B."

The paper deals with practically all the usual troubles.

A. H. N.

**353.\* Cleaning Problems of the Oil-Fields.** R. W. Mitchell. *Oil Wkly*, 30.11.42, 107 (13), 24.—The desirable factors to be considered in a cleaner are: It should be safe to handle and to use from the workers' viewpoint. It should also be safe to metals and materials of construction. Both the cleaner and cleaning method should be as simple as possible, with minimum dependence on technical supervision. The method and material should give speedy results. The cost of cleaning from the overall point of view should be kept as low as possible. Adequate cleaning routines almost invariably lead to longer service life of equipment and apparatus, but the investment has to pay dividends in order to justify itself. Hence final cleaning results obtained at such high costs that the benefits accruing therefrom cannot offset cleaning expenses are of no real value.

The clinging, greasy, oily dirt of derricks and derrick floors is hard to remove effectively and economically. Yet cleaning is necessary, not only for ensuring safe operating conditions, but also to prepare for protective painting whenever required. A new emulsifiable solvent cleaner recommended for this operation is sold as a concentrate, mixed on the job with kerosene or safety solvent, one part cleaner to eight parts solvent to make the cleaning solution. Derricks and derrick floors are readily cleaned with this solution, without need for steam or any source of heat. The cold mixture is sprayed on all surfaces to be cleaned, using a portable spraying tank with suitable nozzle. It is then allowed to soak in for about 15 min., after which all surfaces thus treated are flushed off with cold water under pressure. This completes the cleaning operation. No scrubbing is necessary in 90% of the jobs.

Where dirt accumulations are heavy and of long standing, the initial treatment with the cleaner will remove most of the dirt, but it may be necessary to repeat the treatment, accompanied with scoring or scratching of thick deposits during the soaking-in period, to ensure speedier penetration of the cleaner. More than two applications are rarely necessary—and in fact on most derricks the single treatment is completely satisfactory. This method of cleaning gives very clean surfaces with a minimum expenditure of time and labour, and they can be painted when dry with no further cleaning treatment. In addition to the convenience and economy of working perfectly in cold solution without need for heat, steam, or steam-guns, the solution itself is non-poisonous, non-flammable at ordinary temperatures, and completely harmless not only to the human skin, but also to all metals and other materials of construction.

The cleaning of several important parts of an oil-field equipment is discussed in detail under separate headings. Finally the cleaning of the worker's hands is discussed. The most effective hand-cleaner is one that contains a properly balanced alkaline cleaning material and suitably graded and selected pumice. When the right degree of alkalinity is provided, the pumice works as an effective scouring agent to remove the embedded dirt particles which are unaffected by ordinary soaps. Sand and other harsh abrasives must be avoided, and at the same time too mild a scouring agent, such as corn meal, should not be used for oil-field hand-washing, since such are not strong enough to remove the kinds of dirt encountered. On many other industrial hand-cleaning operations, the vegetable-type scouring agent is completely satisfactory.

A. H. N.

**354.\* Shallow Wells to be Drilled Without Water Casing-Strings.** Anon. *Oil Wkly*, 30.11.42, 107 (13), 36–38.—Complete operating drawings, plans, engineering specifications, and full procedural directions covering methods for drilling of wells with a contemplated saving of about 85% in steel have been released to the oil-producers of California. The procedure will be tried out immediately on six wells in the Wilmington field of California, and it is understood that two large companies operating in Texas also plan to test it. The proposal is not looked upon as a cure-all, as it will probably work successfully in only a portion of the drilling operations. It will be



tried on shallow wells, and it is in this classification that it has probably its best chance of success. Diagrams explaining the scheme are given.

The cost of drilling a 3000-ft. well in California by the new procedure is estimated not to exceed \$12,500, compared to a cost of \$35,000 for a 3000-ft. well three years ago. A year ago, Continental Development Corporation drilled seven wells in the Wilmington field at a cost of \$20,000 each, which then appeared to be a record low cost for such wells in this area.

A. H. N.

**355.\* Drilling Fluid Technology. Part 2.** L. C. Uren. *Petrol. Engr.*, November 1942, 14 (2), 43.—In this second part of the paper the function of the mud as a heat absorber and lubricant for the drill-pipe is discussed. The following sections deal with the physical properties of drilling fluids, density, viscosity and colloidal properties, hydrogen ionization value ( $pH$ ), thixotropic properties, sand and salt content, and with mud-control laboratories. These studies are followed by discussions on the constituents of muds and drilling fluids, dealing with clays, weighting materials, chemical reagents, and special oil-base fluids. The paper ends with a discussion on possibilities of future research. Little has yet been done in exploring the factors that control the efficiency of drilling fluid in its function of lifting drill cuttings. There would be fewer cases of "frozen" drill-pipe if we had a better understanding of the conditions that influence the lifting capacity of the circulating fluid. There is little information of bottom-hole temperatures in drilling wells and the influence of rate of circulation and mud properties on control of such temperatures. More should be learned of the drilling-fluid properties that promote "gas cutting," and better methods of separating entrained gas from gas-cut muds should be devised. There is opportunity for a broad study of oil-base muds, of methods of developing desirable properties in drilling fluids of this type, and of controlling these properties to meet changing conditions. The properties of oil-water clay mixtures is an almost virgin field.

Better methods of quickly evaluating clays for use in drilling fluids should be devised. Is the rate of hydration a significant index in this connection? How may one determine the active colloidal content of a clay or drilling fluid? Of what significance is the size-distribution of clay particles, and can some simple test for determining this be devised? There is much to be learned in the realm of chemical control of drilling-fluid properties. Certain types of gums and resins produce interesting effects. More positive ways of dealing with saline-water contamination of drilling fluids must be developed. Methods of adapting the qualities of the mud to the peculiarities of the formation in which the drill is working—in "heaving shale", for example—present definite possibilities. Successful methods of selectively removing clay sheaths from oil-wet and water-wet formations would be of great value. Tests and instruments should also be standardized.

A. H. N.

**356.\* Modern Technique in Oil-Well Cementing.** P. E. Hilton. *Oil Gas J.*, 3.12.42, 41 (30), 57. *Paper Presented before American Petroleum Institute.*—To place a cement plug opposite an oil-sand so that a water shut-off job is effected, a tail pipe, made of drillable metal, is run in on the bottom of the float or guide-shoe. A float-shoe with or without side ports may be used. When a float-shoe without side ports is used, all the cement is discharged through ports in the bottom of the tail pipe. In the case of a float-shoe having side ports, the cement is discharged simultaneously through the shoe and the bottom of the tail pipe. After cement sets, the shoe is drilled out with the cement plug to a short distance below the shoe for a water shut-off test, the remainder of the plug sealing off the productive zone during the procedure. With a satisfactory cement job assured the rest of the plug and tail pipe are drilled out. It is considered good practice to run a casing scraper between the bit and drill-collar to remove the sheath of cement left by the bit on the casing wall. A clean wall assures positive grip for slips and a smooth surface against which the packing member of testers and similar devices can seal, and prevents cementing tools, which are normally run with close clearances, from hanging up.

The procedure of running and cementing a blank liner in one operation involved the use of a float-shoe having side ports and left-hand square threads (positioned in an upper extension of the valve cage), a square, left-hand threaded sub which screws into the left-hand threads in the shoe, and a circulation or expansion joint, made up on the bottom of the running-in string, which may be either tubing or drill-pipe.



Cementing of the liner, which is run in on the bottom of the string, is performed in the usual manner, after which the running-in string is lowered a few inches, which telescopes the circulation joint and takes the strain off the sub. The running-in string is then turned to the right, which disconnects the left-hand threads, and raised several feet, so that any excess cement which may have come over the top of the liner can be back-scuttled. It then can be removed from the hole, leaving the liner cemented in place with the inside clean and ready for gun perforating.

Cementing liners with perforations below and blank above and a liner off bottom is accomplished by variants of this procedure. Multiple-stage cementing is also discussed.  
A. H. N.

**357.\* Drilling-Rig Functions Logged by Automatic Instruments.** H. F. Simons. *Oil Gas J.*, 10.12.42, 41 (31), 41-43.—The drilling industry is in a transition stage from having practically no instruments to one of complete logging of all the primary functions. The objective is to record the information which makes possible the saving of time and money. This article describes the automatic logging of thirteen factors affecting drilling operations. These include the weight on the bit, revolutions/minute, torque on the drill-stem, mud pressure, rate of penetration, the level of the mud in the pit, the time required to circulate the fluid from the bottom of the hole to the surface, the oil and gas content of the mud, the  $pH$ , conductivity, viscosity, and specific gravity. The trailer housing the unit also includes equipment for other mud tests and core-analysis work.

The arrangement used is unique in that standard rig instruments are connected into the recording instrument. In other words, the regular weight indicator used at the rig is not replaced, but its reading at specified time intervals is recorded. The same is true of the pump pressure, torque and rotary speed, and the rate of penetration. Special instruments were developed for the automatic recording of viscosity and specific gravity of the mud and the delay time (time of circulation of the mud-stream). The torque is obtained from steam pressure in the engine manifold, the revolutions/minute of the rotary from a tachometer, and pump pressure is obtained from the mud-pump pressure gauge, but a stroke counter is to be added, rate of penetration is recorded by the selsyn motor method, the gas percentage in the mud is taken from a sample run into the trailer, viscosity of the mud from an automatic viscosimeter, specific gravity from an automatic and continuous weighing device, and the  $pH$ , oil content, and conductivity of the mud are obtained from a small sample stream.

All the data are recorded on two Micromax instruments. The one instrument records rate of penetration, weight, revolutions/minute, torque, pump pressure, and depth of fluid in the pit. The other instrument records the mud conditions, including percentage of gas, parts/million of oil,  $pH$ , conductivity, viscosity, specific gravity, and delay time. The instruments are driven by current from a small generator direct-driven by a small engine. The travelling chart is the usual ten divisions to the inch, the vertical scale being time, and the horizontal scale being required units for measuring each particular function. A complete recording cycle is made each 2 min. by the instruments. The travelling pen makes a dot on the chart, showing the reading of the instrument for determining one value, and then moves to another position for recording a different factor. On arriving at its new position, it is allowed 20 sec. to assume a stationary position. After this time delay a mark is made, and it moves to the proper position for recording the next factor.

After it has recorded all six or seven of the various functions, it is allowed 40 secs. in which to return to the original starting-place and repeat the cycle. Typical charts are studied and the significance of the tests is explained.  
A. H. N.

## Production.

**358.\* Collecting and Examining Subsurface Samples of Petroleum.** P. Grandone and A. B. Cook. *U.S. Dept. of the Interior, Bur. Mines Technical Paper*, 629, 1941.—The Bureau of Mines has developed a specialized technique for obtaining samples of petroleum under pressure from the bottom of wells, and for subsequent laboratory examination of these samples. This paper discusses the various factors which affect subsurface-oil samples prior to and during sampling. It also embodies a description



of a self-closing subsurface-oil sampler designed to trap the liquid phase of the well-fluid as it exists at the sampling point in the well and to retain the fluid intact until it can be transferred to other apparatus for laboratory examination. Physical properties of subsurface oil are considered, and laboratory methods employed for examining samples are described in detail. Specifically it is stated that the following data are desired by engineers in solving the various problems which arise in the production of petroleum from natural reservoirs: saturation pressure of the reservoir oil; compressibility of the liquid phase at reservoir temperature; total quantity of gas dissolved in different oils at different pressures and temperatures; quantity of gas liberated from solution in oils on decrease of pressure; contraction of liquid resulting from liberation of gas from solution in oils or on reduction in temperature; density and specific volume of the gas-saturated liquids; and pressure-volume relationships of the oil-gas mixtures.

At the time of sampling, auxiliary data relative to the well should be recorded. These include the name, depth, and thickness of the producing formation; sampling depth; oil-producing rate; producing gas/oil ratio; static and flowing pressures and temperatures at the sampling point; well-head blowing pressure; separator pressure and temperature.

Three definite procedures are available for the examination of subsurface-oil samples. These are: differential gas liberation; flash-gas liberation; and determination of pressure-volume relationship. Usually data obtained by the flash-liberation procedure are more applicable than differential-gas liberation data to the physical changes which take place in the gas-oil mixture as it is produced through the flow-string under changing conditions of pressure and temperature. Data from the differential procedure, however, probably represent more closely the physical changes which take place in the reservoir oil remaining in the formation as depletion progresses. Determination of pressure-volume relationship on a sample of subsurface oil provides data for computing the energy available per unit weight of fluid as it undergoes pressure, volume, and temperature changes.

H. B. M.

**359. Steady Flow in the Transition Length of a Straight Tube.** H. L. Langhaar. *J. Appl. Mech.*, June 1942, 9 (2), A-55-A-58.—By means of a linearizing approximation, the Navier-Stokes equations are solved for the case of steady flow in the transition length of a straight tube. The family of velocity profiles is defined by Bessel functions, and the parameter of this family is tabulated against the axial co-ordinate in a dimensional form. Hence, the length of transition is obtained. The curves give a comparison of the author's calculations of the velocity field with those of other investigators, and with the experimental data of Nikuradse. The pressure function is derived from the computed velocity field by means of the energy equation, and the pressure drop in the transition length is defined by a dimensionless constant,  $m$ , which is computed to be 2.28. A discussion of this constant is given in the conclusions. It is to be noted that in viscometry the constant used for the kinetic energy correction,  $m$ , is half this value, due to incorporating in it the  $\frac{1}{2}$  of the energy equation  $KE = \frac{1}{2}\rho v^2$ .

An interesting result is that the inlet length—that is, when the axial velocity approaches the theoretical value of twice the mean, actually 1.98 the mean—is 0.23  $R$  number of radii is attained, where  $R$  is Reynolds number. Nikuradse's data appear to confirm these calculations.

A. H. N.

**360. Graphical Solution of Fluid-Friction Problems.** E. S. Dennison. *J. Appl. Mech.*, June 1942, 9 (2), A-82-A-84.—It is customary to present fluid-friction data in the form of a diagram to log scale in which friction coefficient appears as a function of Reynolds number. Such data are widely applicable to physical circumstances other than those which pertained to the original experiments. The present paper describes a graphical procedure for utilizing data of this character, where analytical methods are not practicable, and resort is made to trial-and-error methods. Similar methods to that described may be found useful in other fields than that of fluid-friction, provided the experimental data are capable of being represented in non-dimensional form.

The paper deals specifically with the case of the fall of spheres in fluids and with the ordinary pipe-flow problem; but the method is general to all similar problems. The value of the method lies in that a direct solution is possible for problems where the



factor sought is included on both ordinates, such as the velocity or the diameter in pipe-flow, where these factors enter both the friction factor and the Reynolds number. Both the derivation and illustration of the analyses are given. A. H. N.

**361.\* Influence of Extraction Methods on Ultimate Recovery.** S. F. Shaw. *Oil Wkly*, 2.11.42, 107 (9), 28.—The paper is a comprehensive and balanced review of theories and facts about ultimate recovery of petroleum from sands, and should be read *in extenso* to obtain its full value. Spacing of wells is discussed at length with the conclusion that, economics being first considered and found favourable, close spacing would result in greater recovery ultimately than wide spacing. The statement is supported by much data. For instance, the Turner Valley field, Canada, with a very tight reservoir, is one in which spacing has been on basis of 40 acres/well. The results to date seem to the writer to indicate that the yield will be perhaps 50% of what would have been the recovery had the spacing been on a basis of one well to 10 acres. However, with the prevailing price for oil and with cost of drilling amounting to between \$150,000 and \$200,000/well, it would not be profitable to drill four wells to double the recovery that appears likely to be obtained from one well. In this connection it will be interesting to observe the results obtained later in such fields as Wasson, Slaughter, Magnolia, and other similar limestone fields which have been developed on basis of 40 acres to the well.

The main conclusions of this long paper are: (1) Economics in the production of oil require greater consideration than ultimate recovery. (2) Ultimate recovery in many fields is increased by close spacing and production at maximum practicable capacity. It is possible that in nearly all oil-fields this conclusion may apply, but, if correct, it can become evident only in the course of time. (3) Acidizing, shooting, and completing wells with large casing aid in enabling wells to be produced at high capacity. (4) More energy in gas associated with the oil is available for moving oil through the sand from a given quantity of gas when expanding through a given range of lower pressures, than when expanding through a given range of high pressures. (5) Secondary-recovery methods are very important in increasing the recovery of oil where primary methods have not resulted in high-percentage recovery. When primary methods have resulted in high ultimate recovery it is a question as to whether secondary methods are economically applicable. (6) Secondary methods of artificial water-flooding seem to yield higher ultimate recovery when production is at a high rate. If this is a correct conclusion, it would appear as if natural flood with high-capacity production would yield higher ultimate recovery than when production is at a low rate.

The paper is well illustrated with tables and a graph, and twenty-nine selected references are appended. A. H. N.

**362.\* Many Avoidable Production-Equipment Failures Due to Field Abuse and Neglect.** Anon. *Oil Wkly*, 9.11.42, 107 (10), 46.—The paper details various methods for maintaining equipment for a long time, in particular, stressing proper lubrication. A film of lubricant of sufficient strength to withstand localized contact pressure must be provided at all points of moving contact to prevent wear. When speeds are high, contacts are usually of short duration and loads are light, requiring the selection of a light-bodied lubricating oil. When speeds are low, contact time is longer and loads are heavier, requiring a comparatively heavy-bodied oil for proper lubrication. When loads are exceedingly high/unit of contact surface, extreme pressure lubricants are best suited for minimizing wear. In all cases it is advisable to use only high-grade lubricants.

Because it is difficult to keep containers clean on a drilling well or an oil lease, all oils and greases should be supplied in 4-, 5- or 6-quart sealed containers. This is particularly advisable in the case of all greases and oils that must be used in force-feed lubricators. Temperature affects the viscosity and lubricating properties of mineral oils, necessitating the use of different types of oil in summer and winter. At atmospheric temperatures below 55° F. an oil of lower viscosity should be used than is ordinarily recommended for atmospheric temperatures above 55° F. The maximum oil temperature at which standard lubricating oil should be used for bearing surfaces is 180° F. Particular care in the selection of lubricating oil is essential in the use of the many machines where the same oil is used for various parts, such as bearing,



slides, and gears. If an oil of high viscosity and high pour test is used in such a machine at low atmospheric operating temperatures, the oil will not flow readily and bearing failures may result. Methods of applying the lubricants are detailed.

The paper further deals with starting batteries, enclosures for equipment, and methods of rebuilding equipment or salvaging it. A. H. N.

**363.\* Maintenance Department Features Many New Tools and Efficient Techniques.** G. M. Wilson. *Oil Wkly*, 9.11.42, 107 (10), 51-60.—The activities of this department come in contact with nearly every phase of the producing and drilling divisions of the company, and though there is, of necessity, a certain amount of overlapping, reconditioning work is performed in at least five major subdivisions of that department. These are: (1) valve and fitting shop, where all types and sizes of valves, fittings, and rotary chain are overhauled, as well as metal spraying and some small lathe work; (2) instrument and meter shop, where all kinds of gauges, recording meters, regulators, drilling instruments, etc., are repaired and rebuilt; (3) machine shop, which handles the repair and maintenance of larger equipment, such as pump cylinder re boring, engines, clutches, and similar heavy equipment; (4) tubular-goods handling section, which has developed some very practical measures in the salvaging and extension of life of such materials; and (5) system of records which facilitates inter-departmental "buying" and "selling" and keeps a running inventory of all equipment that comes through these departments. The various subdivisions are described in detail, the valve section being given particular attention.

An interesting section is the metal-spray rebuilding section. The operation, briefly, involves the forceful spraying of atomized molten metal on to the previously prepared bonding surface of the object being built up. Ordinarily, the object—in this case, a gas-engine piston—is chucked in a lathe, and the gun is mounted on or beside the tool-mount where it can be made to travel slowly back and forth, gradually building up the surface of the parent metal. Of prime importance—and the success or failure of the job depends on it—is the proper preparation of the parent metal surface. This involves taking a very coarse, rough cut on the surface, using a tool that literally tears the metal off, leaving a series of rough, jagged ridges and grooves. Since the bond of metal spray is a mechanical one, the jagged surfaces allow the impinging metal particles to take a firm grip on the base metal. After the surface has been built up sufficiently oversize, it is ground down with a power grinder, also mounted so that the tool-feed will carry it back and forth across the surface.

A steel plug is forced into the skirt-end of the piston to give strength and "body" to the latter. This plug should extend beyond the skirt-end far enough to permit the lathe jaws to take a full grip on it and allow sufficient clearance between the face-plate and gun when the latter gets to that end. This extension also keeps the piston skirt away from the face-plate so metal spray will not be so likely to get on to the face-plate or the dogs. A. H. N.

**364.\* Simple Preventive Maintenance Rules Prolong Sucker-Rod Life.** G. M. Wilson. *Oil Wkly*, 9.11.42, 107 (10), 113.—When it is realized that an average string of sucker-rods may be subjected to loads ranging from 10,000 to 30,000 lb., and suffer as many as 15,000 to 30,000 reversals of motion/24 hr. day, it is apparent that the much-neglected sucker-rod really has a difficult job to perform. With a small nick or scratch already established in one of the rods, it is easy to see how a full break can soon develop when the string is being subjected to a round-the-clock series of alternate stretching and contracting under stress of the well load. When this load varies or becomes more severe due to a gas-lock or pumping-off conditions causing frequent pounding in the strokes, final parting of the rods at this zone of weakness will be materially hastened.

Wells which are not provided with derricks offer one of the best opportunities for rods to become damaged. On such wells the rods usually have to be laid down on the ground or some makeshift supporting blocks, the latter quite commonly being only several short pieces laid at infrequent intervals along the ground. Rods lying thus along the ground, particularly if the well is down for a prolonged time, are an invitation to trouble. Dirt, abrasives, use of hammers, etc., are added sources of trouble. The use of racks, proper haulage, and of protectors is urged, together with the keeping of records. One of the simplest and least expensive means of overcoming



such problems, which arise every day, particularly in the larger companies, consists in the use of a tagging system. Each crew foreman carries a quantity of two-section, perforated tags, on each section of which he jots down, in places provided for such data, the pertinent facts relating to that particular rod which he is laying down, and which he feels warrants further checking by the salvage or warehouse department. The upper half of the tag is then removed and tied to the rod before leaving the well. The lower half is retained and later turned in at the office at the time he makes out his daily work report.

For legibility, tags should not be less than about 3 in. wide and 6 in. long, being perforated in the middle to give two cards 3 by 3 in. when torn in half. Representative of the instruction headings which should be printed on each side of the two halves include: (1) The serial number (same number on each half of tag.) (2) Well number and lease. (3) Item reported. (4) Where taken if removed from well premises. (5) Why discarded or laid down. (6) Crew foreman's signature and crew number.

A. H. N.

**365.\* Co-ordination of Conservation Practices in the Various States.** E. G. Dahlgren, *Oil Gas J.*, 12.11.42, 41 (27), 118. *Paper Presented before American Petroleum Institute.*—This paper discusses the co-ordinating activities of the Interstate Oil Compact Commission in the conservation of oil and gas in the various States. The work of the various compact committees is described and the conservation policies and administrative activities in the various oil producing States are summarized.

The legal committee of the Interstate Oil Compact Commission has been doing some outstanding work during the past year. At the 19th December, 1941, meeting of the Compact Commission in Oklahoma City, the legal committee adopted the programme as follows: (1) A study of the suggested model conservation law for the purpose of making any necessary amendments and corrections. (2) A study of the legal phases of repressuring and recycling. (3) Preparation and publication of synopses of all the statutes of the various oil-producing States. (4) A study of the relationship between the Federal Government and the States concerning the legal aspects of oil and gas conservation. (5) The preparation of a skeleton statute that might be advisable in non-producing States or in States with small production.

The paper deals separately with the conservation laws and practices of each State, including those which are only potential producers.

A. H. N.

**366.\* Present Allocation Practices.** D. R. McKeithan. *Oil Gas J.*, 12.11.42, 41 (27), 154. *Paper Presented before American Petroleum Institute.*—The development of allocation methods in the various oil-producing States is reviewed and analyzed; also a brief description of the administration and mechanism of the systems employed is presented. Conclusions reached therefrom are: (1) The oil industry is operated primarily on a competitive basis, and this factor adds to the complexity of the attainment of maximum efficiency in conservation, and allocation is extremely difficult. (2) Systems of control in the various States are geared to meet local operating conditions, as well as background and temperament of the people, in conformity with American standards of localized governments. (3) The actual mechanics of allocation used in the various States differ greatly and, further, it appears that one basic law or one rigid formula cannot be applied reasonably to all the varied conditions.

Vast strides towards general improvement have been made in recent years; however, there is still room for considerable betterment; and the "Standards of Allocation of Oil Production Within Pools and Among Pools," as set out in the report of the American Petroleum Institute's Special Study Committee on Well Spacing and Allocation of Production, should be of material assistance in further progress.

The paper deals with the chief oil-producing States individually under separate sections.

A. H. N.

**367.\* Evaluation of Effective Displacement Pressures for Petroleum Oil-Water Silica Systems.** F. C. Benner, C. G. Dodd, and F. E. Bartell. *Oil Gas J.*, 12.11.42, 41 (27), 199. *Paper Presented before American Petroleum Institute.*—The relative wettability of reservoir rock by crude petroleum oils and by water appears to constitute a more complex problem than has hitherto been generally assumed. Certain constituents of the



oils, even though present in minute amounts, may greatly influence the degree of wetting of the solid by the oil. In this investigation, an evaluation of relative wetting of reservoir rock by reservoir fluids has been attempted by the measurement of contact angles formed by petroleum oils and water on solid silica plates. Displacement pressures calculated from the contact-angle data were found to be in reasonable agreement with displacement pressures actually observed with the petroleum oils and water in packed-silica powders.

A study was made of both "advancing" and "receding" contact angles for a series of petroleum oils and water on silica. Advancing interfacial contact angles were found to vary over a wide range for the different systems studied, in some instances being less, and in others considerably greater, than  $90^\circ$ . Were the advancing angle alone to be used as a criterion of wetting, it would appear that in some systems water wets silica to a greater degree than does oil, but in other systems the converse is true. It was found that receding interfacial contact angles of the systems observed invariably assumed values of less than  $90^\circ$ . By this criterion, silica appeared to be better wetted by water than by oil. The advancing interfacial contact angle is believed to be a better indication of the hydrophilic or hydrophobic nature of the reservoir material containing the oil than is the receding angle.

The "hysteresis" effect was very pronounced in nearly all the systems studied. For most of the systems, the silica appears to have dual wetting characteristics, inasmuch as the receding contact angle is less than  $90^\circ$ ; whereas the advancing contact angle is greater than  $90^\circ$ . The results from this research indicate that spontaneous displacement of oil from the underlying solid by water should occur only in those cases where both the advancing and the receding angles are less than  $90^\circ$ ; that spontaneous displacement of water by oil should occur if both angles are greater than  $90^\circ$ ; and that no spontaneous movement should occur where the two angles are on opposite sides of  $90^\circ$ .

It appears that, in the majority of cases, measurement of the contact angles formed on a silica plate between a petroleum oil and water will serve as a fairly reliable guide for the determination of the displacement tendencies of oil by water or of water by oil. The results obtained give fairly definite proof that crude petroleum oils contain constituents which become adsorbed on siliceous surfaces, causing those surfaces to assume hydrophobic properties.

A. H. N.

**368.\* A Review of Multiple-Zone Well Completions.** I. W. Alcorn and W. A. Alexander. *Oil Gas J.*, 12.11.42, 41 (27), 224. Paper Presented before American Petroleum Institute.

—This paper presents representative data on multiple-zone completions which have been made in the several oil-producing regions of the United States. It is a joint effort of the topical committees on drilling and production practice, and constitutes a progress report on the subject. The desire of the oil industry to find ways and means of conserving steel in its drilling and production activities led to the decision to undertake this survey. An analysis of eighty multiple-zone completions indicates that an average per-well saving of 105 tons, or 4%, was realized in a multiple-zone completion, in comparison with two standard-well completions.

Available information indicates that the initial completion of wells in more than one zone can be accomplished successfully. Reliable data on the effectiveness of the method are at present limited to the earlier stages of multiple-zone production. Sufficient representative data on the later life (especially during the artificial-lift period) of multiple-zone wells are not yet available to permit drawing any definite conclusions as to the ultimate over-all practicability of the method. Existing conditions with respect to critical materials may reasonably be expected to result in an accelerated development of improved technique and more advanced methods of application.

A. H. N.

**369.\* Conditioning Oil-Wells for Secondary Recovery.** F. R. Cozzens. *Oil Wkly*, 16.11.42, 107 (11), 40.—The paper describes in brief certain treatments of wells. An oil-and-soap solution forms the basic treatment in Eastern areas, and the process is put into effect when the wells can no longer be pumped at a profit. While the pumping equipment is still intact, the reservoir or shot-hole at the bottom of a well is filled nearly to capacity with live oil into which has been stirred soap chips or granulated soap, in proportion of 5 lb. of soap to each barrel of oil. Six to ten



barrels of this solution are generally needed for each well. The lead line in the tubing is next connected with the gas-vent in the casing-head, and the well is circulated continuously for a period of 36-48 hr., then pumped dry. In the pumping out process, the oil-and-soap solution is conducted to a tank, where, after the residue has settled out, the oil is drawn off, mixed with about the same amount of soap as before, then again introduced into the well for another circulation. After two or three intermittent circulations of this kind, during which a careful observation is made as to type of residue pumped out, the large majority of wells show an increasing amount of live oil and gas. This is proof that the face of the reservoir has been washed clean and old channels opened.

Uses of paraffin solvents, acids, and shooting are briefly discussed. A. H. N.

**370.\* Tracer Gas Used to Check Repressuring Progress.** H. F. Simons. *Oil Gas J.*, 19.11.42, 41 (28), 36.—The injection wells are equipped with packers so that the input into each horizon can be controlled. The producing wells are not equipped with packers, and produce through a common string of tubing. By injection of the tracer gas into one of the formations, the time for movement of the gas between the input and producing well can be determined. When it is desired to test a well, approximately 600 cu. ft. of 70% tracer gas is injected along with the repressure gas. This amounts to about 500 p.p.m. of the tracer in the injected gas. Gas from the producing well is then checked until evidences of the tracer begin to appear, build up, diminish, and finally disappear. The shortest time taken for the tracer to show up in the producing well was only a few months in one case, which showed there was evidently channelling of the formation. Injection in this particular case was discontinued temporarily.

The amount of tracer in the produced gas ranges from 3 p.p.m. to 175 p.p.m. Practically all of the injected tracer is recovered from the reservoir. The tracer gas used on these tests is manufactured in a small building, located at one of the compressor plants, at Loude. The tracer gas is generated from carbon dioxide and charcoal. The plant has a capacity of 600 cu. ft. of 70% pure tracer gas with two reactors in series operation/day at atmosphere. It is bottled at 900-lb. pressure. The procedure is for the test crew to take three bottles of gas out in the morning and three in the evening. These are connected to a bypass at the injection wells and the bottles are then purged of the tracer gas. The cost of the tracer gas is approximately 6 cents/cubic foot. A. H. N.

**371.\* Long-Stroke Pumping Unit Uses Mine-Hoist Principles.** N. Williams. *Oil Gas J.*, 19.11.42, 41 (28), 50.—In the design of the unit, which, so far, is limited to working models, application is made of an old mine-hoisting principle, that of variable leverage as supplied by conoidal drums, to eliminate the unbalanced condition that exists at the end of the pumping stroke. Although the idea and use of conoidal drums for hoisting are far from new, this is the first time, it is claimed, that any attempt has been made to apply the principle for lifting fluid from beneath the earth's surface. Variable leverage of a conoidal drum is obtained through the changes in its effective radii. It is this that gives the constantly changing counterweight effect in the operation of the new pumping unit, and provides the unbalancing action at the completion of each stroke to start, or help start, the succeeding stroke while the prime mover is in process of acceleration. The change in radius which controls the length of stroke and speed can be governed by the size of the cable reels, and the thickness of the cable winding thereon, and by the steepness of the conical grooved drum. Elimination of the unbalanced condition at the end of a stroke makes practical the use of a simple prime mover in continuous frequent-reversing service.

Essentially the new unit includes an automatic reversing controller for a prime mover; a speed-reducing mechanism operated by a drive connection from the prime mover; an operating shaft from the speed reducer; the conical drum, secured on the shaft, and on which is wound the cable or chain operating the pump-rod, and the drum, on which is wound the line operating the counterweight. The arrangement of these elements is shown in accompanying drawings. Details of counterweight arrangements are given. Pumping strokes of from 50 to 75 ft. are considered feasible with the unit, the length of the stroke being limited only by the size of the drum and the height in the derrick of the blocks through which the pumping and counterweight



lines travel. With strokes of this length there is not only a substantial reduction in loss of energy due to rod stretch as contrasted with the loss incurred in the usual stroke lengths of from 2 to 6 ft., but also the less frequent stretching involved in the long strokes results in much reduced rod fatigue.

Other advantages claimed for the long-stroke unit are: (1) Elimination of gas-lock by winding the plunger out of the barrel to release the gas. (2) Elimination of paraffin from centre of tubing by rod-joints which will pass all points in the tubing. (3) Loosening of sanded plungers by a jar in the rod-line actuated by reversing the prime mover to produce the jarring action. (4) Reduction in cut oil and less disturbance of sand through the elimination of short, choppy strokes. (5) The unit can be equipped to shut down automatically when a well pumps off or starts to flow. (6) Slow rate of acceleration of the stroke reduces peak well weight. A. H. N.

**372.\* Progress in Reservoir Technology. Part 4.** M. Muskat. *Petrol. Engr.* November 1942, **14** (2), 65.—Well spacing is the first item discussed in this part. It is argued that from a physical viewpoint there is no basis for preferring close to wide well spacing or vice versa. It is from the practical and financial standpoint that there is a basis for distinction. Even should there be a tendency for the economic ultimate recovery to increase somewhat with the well density, this in itself does not provide the answer to the well-spacing problem. For obviously it would be economically unsound to increase the well density if the increased production before abandonment would not pay for the cost of the additional wells. Indeed, it is only by balancing the added value of the production against the increased investment and operating costs that the true economic optimum well spacing may be determined. In particular, we must look for the maximum in the difference between the curve for the income from the oil recovered and the cost of the development and operation, each plotted as a function of the well spacing. In determining each of these, account must be taken of the effect of the well spacing on the life of the operations and the interest charges on the investment and discount rates for future profits. It is thus found that the curve for income from oil recovery rises rapidly at first and then quickly flattens out as the well density increases. On the other hand, the cost curve maintains an approximately linear rise throughout the whole range of practical spacing variations. The optimum spacing—that for maximum profit—is then the abscissa for maximum difference between the curves, or for equal slopes. For physical and cost constants of such magnitudes as are encountered in practice, this method leads to optimum spacings for gas-drive fields as are actually indicated by direct field experience—namely, 20–100 acres/well.

Proration is next discussed. From the technical standpoint, proration has had two major effects. First, as implied by the definition of the term, it has forced the operation of wells and fields at lower production rates than possible by wide open flow. Second, it has created the problems of proration formulæ, and allocation of production, which, in turn, have stimulated much of the progress already discussed. These two effects are detailed.

A brief study of conservation concludes the paper.

A. H. N.

**373.\* Oil Recovery by Under-Pressuring.** F. R. Cozzens. *Petrol. Engr.*, November 1932, **14** (2), 72.—Under-pressuring begins after a secondary-recovery method has been in effect for some time. The method constitutes removing the false bottoms of certain of the intake wells, and lowering the air-induction tubing so that the extreme low sections of the sand will receive the repressuring medium. To confine the pressure, packers are usually run and set in the string immediately below the areas of sand that have been previously pressured. There is no methodical rule regarding the number of wells to be thus treated, but most operators use a ratio of one-third their intake wells, situated at various points over the field. When the estimated number of wells have been deepened and recompleted, air or gas is forced into them at full capacity, and as fast as the sand will take it. As pressure builds up, the pressure on the remaining input wells in the upper sand zones is gradually reduced until only sufficient pressure is maintained to push the oil to producing (open) wells. Pressure to the deepened wells, however, is continuous, and a built-up pressure of 300–500 lb. is necessary for best results.

An air-pressure increases in the base of the sand, the fluid content (oil, water, and



other drainage) expands and rises. The oil, being lighter, is first to enter the producing sand zones, and is later followed by water. A good percentage of oil is generally recovered before water becomes troublesome in the pumping wells, but even as water increases in volume, the operator still benefits. The rise of water in the sand floods the channelled-out portions and acts as an effective remedy for the former bypassing of air. The increasing water volume likewise has a tendency to release considerable oil from side margins of the sand, and to float oil from crevices and pockets that cannot be reached by air- or gas-pressure alone. Combined pressure (air or gas and water) from below keeps fluid always within the scope of producing wells throughout the entire life of the recovery project.

A. H. N.

**374.\* Thermo-Siphon Heaters for Oil-Field Operations.** H. Vance and E. G. Smith. *Petrol. Engr*, November 1942, **14** (2), 103.—The most common application of the thermo-siphon heater is in the flow-tank or gun-barrel for the purpose of breaking down emulsions. In a gun-barrel installation, where the oil to be heated is floating on top of a body of hot water, the area of contact is equal to the cross-sectional area of the tank containing the two liquids. Thus, we find that the diameter of the tank is the controlling factor, provided that the difference in temperature between the oil and the water is the same. In order to increase the surface of contact one may install in the ordinary gun-barrel what the writers have termed an emulsion pre-heater, which consists of coils very similar in design to steam-coils used in heating oil with steam. The oil from the well passes directly through these coils, which are in turn covered with hot water; then the oil is discharged through an enlarged pipe section into the body of the oil itself. This problem of surface contact is directly responsible for the low heat transfer possible in a small oil and gas separator.

Complete design data are presented and used in typical examples. A. H. N.

**375.\* Gun-Perforated Well Completions.** B. C. Craft. *Petrol. Engr*, November 1942, **14** (2), 112.—The study was made to determine the optimum number of perforations/ft. of casing as a guide to engineers and operators. Theoretical work, based on Muskat's formulæ, is presented. Experiments showed the pressure loss resulting from the sand bridge over rectangular slots to be small, if the slot width used is equal to twice the grain size at the 10 per centile, but to increase as the slot width is reduced. Likewise, it is believed that the effect of bridging or filling of the perforations with sand would be small. Although the pressure loss, due to openings in the slots, has been found to be small compared with the convergence loss, it would be approximately twice as large as when the liner is set in open hole, due to the orifice effect of the perforations. If the total equivalent area of the perforations is twice that of the openings in the liner, this loss is one-fourth the liner loss; conversely, if this area is one-half, the pressure loss will be increased four times.

It is impossible to estimate how many of the gun perforations remain open. It is standard practice with some operators to run a rock-bit or mill past the perforations before going in the hole with the well-setting to prevent bullets that may have wedged in the side of the casing from tearing the screen and to remove the mud filter-cake over the perforations. Other perforations may be plugged with small pieces of cement and shale. Evidence of plugging has been obtained from an examination of screens removed from high gas-oil ratio wells. The screen-pipe either showed the effect of, or was cut through entirely by, the jetting action of sand particles flowing through the limited number of open perforations. Due to the high pressure losses resulting from convergence or crowding of the fluid at the perforations, the pressure loss due to the orifice effect of the perforations, the cost of reworking wells when the screen-pipe has become gas cut, and finally, due to the plugging of a number of the perforations, it seems safe to conclude that the combined area of the gun perforations/ft. of casing should be within the range of one and a half to twice that of the opening/ft. of screen-pipe.

A. H. N.

**376.\* Prediction of the Shrinkage of Crude Oils.** D. L. Katz. *Oil Wkly*, 30.11.42, **107** (13), 17-22. *Paper Presented before American Petroleum Institute*.—Recent information on the methods of computing the density of liquids containing dissolved gases makes possible more accurate calculation of phase behaviour under pressure for



crude-oil-natural-gas systems. This report covers several methods for predicting the shrinkage of a reservoir crude oil when such field data as reservoir pressure, reservoir temperature, solubility of gas in the oil, lease-tank crude-oil gravity, and gravity of the gas (air equals 1) are given for the reservoir under consideration. When all of these items are known, the method is straightforward and reliable; whereas if only solubility, crude-oil gravity, reservoir pressure, and reservoir temperature are known, the method becomes less reliable. For cases in which only crude-oil gravity, reservoir temperature, and reservoir pressure are available, even more approximate methods are presented. A rough method for prediction of the solubility of gas in the crude oil from saturation pressure and crude-oil gravity follows the shrinkage calculations.

The shrinkage calculations do not depend on the separation process—*i.e.*, flash or differential vaporization of the gas—because the properties of the resulting gas and liquid, as well as the quantity of gas liberated, compensate for any changes in the shrinkage value. A method of computing the density of crude oil saturated with natural gas has recently been presented. The method consists of computing the density of the liquid at 60° F. and 1 atmosphere of pressure by the use of additive volumes, and correcting the density from 60° F. and 1 atmosphere: first, to the pressure desired, and then to the desired temperature. The densities of all constituents heavier than ethane are taken as the normal density in grams/millimetre or pounds/cu. ft. of the pure constituent at 60° F. and its vapour pressure. For methane and ethane, it was necessary to devise an apparent density chart which gives the density of methane and of ethane as a function of the concentration (in weight per cent) in the liquid, and of the density of the constituents other than methane or ethane. In most cases the method will compute the density of the crude oil saturated with natural gas to about  $\pm 2\%$ .

The calculation of the density of a crude oil saturated with natural gas requires the analysis of the gas which is liberated when the crude oil is reduced in pressure, such as to atmospheric, together with the density of the remaining crude oil at the lower pressure. The quantity of gas liberated is added to the final crude oil, and the total volume of the crude oil saturated with this gas is computed at the saturation temperature and pressure. This volume minus that of the residual crude oil gives the shrinkage caused by vaporization.

The paper is well illustrated by tables and examples.

A. H. N.

**377.\* Policies and Results of Water-Flooding in Kansas.** P. Reed. *Oil Gas J.*, 3.12.42, 41 (30), 31.—Those attracted to water-flooding for the first time frequently under-estimate the time and expense usually consumed in selecting and acquiring producing properties suitable for water flooding. It is now widely recognized that in order to make a profit out of flooding at prevailing prices for crude oil, it is necessary to recover at least 3,000 bbl./acre from sand with considerably more than 35% saturation. In order to do this with the 330-ft. and 440-ft. spacing patterns commonly used, operators have been looking for sand bodies at depths less than 700 ft. containing oil of well above 30° A.P.I. Operators have found these conditions met to a larger extent in South-eastern Kansas than in any other Mid-Continent area. There, the shoestring sands of fairly uniform and moderate permeability without the presence of a water-table are well suited to water-flooding.

In reviewing the first five years of water-flooding, it is seen that, following the initial influx in 1936 and 1937, there was a steady expansion in the application of this technique. However, it was somewhat retarded by difficulties in finding outlets for production. Last year and this year, due to war conditions, there has been a definite acceleration of the trend to water-flooding. Of the thirty-three projects of which records are available, only nine showing a substantial increase were producing more than 100 bbl.

The typical successful water-flood property produced between 7 and 20 bbl. from 80–160 acres before flooding. It has been developed for water-flooding according to a systematic plan which provides for adequate water-treating programme and progressive extension of water-flooding each year to parts of the property which had hitherto been unaffected by the drive. In this way the operating organization can be held together on a fairly permanent basis and the company can maintain production somewhere near the same levels from year to year. Although some operators favour



producing by flowing throughout the life of the property in order to save the expense of pumping, the general practice is to pump during the early life of a property, and flow during the last stages, when the oil-water ratio is greater than 1 to 10. By such a progressive programme it is possible to shift pumping equipment from flowing wells to wells in the newly flooded sections. Furthermore, this plan enables the company to profit by its experience so that more efficient methods may be applied as operations proceed. However, certain operators favour a policy of developing an entire property as rapidly as possible for water-flooding in order that oil may be recovered in a short period of operation with a minimum of overhead.

Details and certain features of special interest in various fields are discussed.

A. H. N.

**378.\* Raising Oil and Water from Oil-Wells.** S. F. Shaw. *Oil Gas J.*, 10.12.42, 41 (31), 51.—A portion of this paper is cut by the Censor from *Journals* coming to England. In the remaining portion the combination air-lift plunger-pump method used in oil-fields is discussed. First trials were unsatisfactory, but a later method was not entirely so. In this method the compressed air was employed to drive the pump engine, and this air was exhausted into the pump column and used to lift the oil by air-lift from the pump to the surface. The use of compressed air to operate the pump was a distinct advantage in that it eliminated the need for a 6500-ft. string of sucker-rods, but the application of the compressed air to drive the pump and also to lift the oil to the surface met with difficulties, in that there was no effective means of controlling the supply delivered to the pump as distinguished from the supply of air required to perform the air-lift operations. In fact, no attention was paid to the air-lift phase of the lifting operation, and yet this was most important, since it involved lifting the oil from the pump to the surface, a distance of nearly 6500 ft. Improvements could perhaps have been made in the type of pumping engine employed, but the main difficulty lay in the improper and misunderstood method of applying the air-lift.

It is believed by the writer that this method of using the plunger pump driven by compressed air in combination with air-lift possesses considerable merit, and should find an important application on oil-field lifting operations, particularly in deep wells. After flowing, pressures in oil-wells decline to a low point, the consumption of gas/barrel of oil increases to an excessive point; also the quantity of oil entering the well is reduced by reason of the back pressure held against the sand face through the use of this excessive quantity of gas. Therefore, if an increased submergence can be artificially created for the air-lift and at the same time the back pressure at the sand face is reduced, there will be an increased production of oil, and a smaller quantity of compressed air required to lift the oil.

A. H. N.

### Transport and Storage.

**379.\* Results of Corrosion Inhibitor Demonstrated in Products Line.** S. S. Smith. *Oil Gas J.*, 24.9.42, 41 (30), 85.—The successful application of a corrosion inhibitor, developed by Dr. Wachter of Shell Development Co., to an 8-in. pipe operated by Shell Oil Co., is described. The inhibitor is very soluble in water, and a solution is injected continuously by means of a special device into the product stream at the pumping station. Concentrations of inhibitor in excess of 0.03% are required in the free water present to preclude corrosion, and the water must be maintained alkaline (pH value in excess of 7.5). To this end caustic soda was added to the injection solution. Care is also taken to maintain the line as water-free as possible. Control of injection may be obtained by testing inhibitor concentration of effluent water. Operations prior to injection had resulted in appreciable scale formation and reduction of line capacity. Charts presented show (1) line temperatures and line gradient; (2) inhibitor injection rate, inhibitor content of effluent water, scale removal, scraper usage, and line capacity (expressed as "c" factor by the modified Hazen and Williams formula) over a prolonged period of operation with inhibitor injection. In the early stages inhibitor injection rate was small but was later increased appreciably with a resulting immediate improvement in "c" factor and in scale removal.

Tentative conclusions reached from the accumulated data are:



- (1) All effects in a long line are much slower than usually anticipated, and only a long-period collection of "c" factors and scale receipts is useful for analysis.
- (2) Over-inhibition is necessary to improve "c" factors and to remove scale.
- (3) Effluent inhibitor concentration is the index of effectiveness of inhibition.
- (4) A products line can be cleaned and maintained clean by the use of this inhibitor, with only nominal scraping to move scale dislodged from the pipe into the stations.
- (5) There is no danger to the line resulting from moderate over-inhibition to hasten scale removal. No difficulty has resulted in ordinary operation from the use of the inhibitor.
- (6) An old scaled line with "c" factors under 130 will probably improve for a year under inhibition and will attain approximately original factors.

The inhibitor is now in general use on all products pipe-lines operated by Shell Oil Co. R. A. E.

**380.\* Mitigation of Underground Corrosion of Pipe-Lines.** Anon. *Oil Gas J.*, 1.10.42, 41 (21), 37.—Information from a review prepared by the Bureau of Standards is presented. Well-drained areas should be selected for tank farms and other projects requiring buried tanks or containers.

Where choice is possible, right of way of pipe-lines should be selected so as to avoid swamps and corrosive soils. Where this is not possible, back-filling the trench with sand or other non-corrosive material, or even with lime, may be advisable. Other precautions recommended are careful tamping of soil around the lower half of the pipe, and avoidance of contact with rocks, sticks, or other solid materials. Corrosion tends to increase with the depth at which pipes are laid.

There is little difference in the rates of corrosion of commonly used ferrous pipe materials. Average results of the Bureau of Standards soil-corrosion tests indicate that wrought iron corroded slightly less, and cast iron slightly more, than Bessemer steel during the first twelve years of exposure, but the relative merits differ for different soil conditions.

Addition of nickel to steel is more effective than chromium, but corrosion is not greatly reduced until the percentages of these materials become sufficient to affect materially the cost of the alloy. An alloy containing 14% Si is very resistant to most soils, but hard to machine and brittle. The increased cost of the more resistant materials must be balanced against cost of leaks to decide whether their use is justifiable. For reasons enumerated, lead coatings have not been generally successful underground.

Some of the more important papers on cathodic protection have been summarized for distribution by the Bureau of Standards. Under some conditions protection of pipes by this method is less expensive than any other means of keeping them in service. Insulated coatings greatly reduce required current consumption, and although data on the life and effectiveness of coatings are meagre and conflicting, a reasonable evaluation of the economics of combined coating and cathodic protection should be possible.

Insulating joints, correctly installed at strategic points along a line, may be of much value in combating corrosion, and advice regarding installation is given. Uses and limitations of wooden and cement-asbestos pipes are described. R. A. E.

## Cracking.

**381. Patents on Cracking.** W. J. Degnen. U.S.P. 2,305,569, 15.12.42. Appl. 15.4.38. High-boiling hydrocarbons are catalytically cracked by an endothermic reaction which involves contacting vapours of the hydrocarbons at high temperatures with a pulverulent cracking catalyst. The heated particles of the solid catalyst are dispersed in a heated vapour stream of the hydrocarbons. The dispersion is passed upwards through a cracking zone of relatively large cross-sectional area in which the velocity of the vapour component is sufficiently low to produce a cloud-like accumulation of catalyst particles. The cracked vaporous products are withdrawn from the upper part of the zone and catalyst particles contaminated with carbonaceous matter



are recovered. These particles are continuously passed to a regeneration zone, where carbonaceous contaminant is removed by combustion.

P. L. Veltman. U.S.P. 2,307,434, 5.1.43. Appl. 7.2.40. In cracking of a hydrocarbon oil, a stream of vaporized feed oil is introduced at a temperature between 800° and 1000° F. into a reaction zone containing a mass of solid, conversion catalyst in particle form. Heated feed-oil vapours are passed through the mass, while an absolute pressure is maintained in the catalyst zone within a relatively narrow range extending both below and above a predetermined mean pressure. The pressure in the reaction zone is subjected to substantially continuous fluctuation within this range, without interrupting the flow of hydrocarbon feed. Pressure is alternately increased and decreased, over a range of about 10-15 lb. Hydrocarbon products are finally discharged from the reaction zone.

K. K. Kearby. U.S.P. 2,307,795, 12.1.43. Appl. 14.12.38. In the catalytic conversion of hydrocarbon oils a bentonitic clay is used as catalyst. The bentonitic clay is pretreated in its raw state with dilute strong mineral acid to remove 50% of its original impurities. Regeneration of the clay is carried out in the absence of a substantial amount of water vapour. H. B. M.

### Hydrogenation.

382. Patent on Hydrogenation. J. Turkevich. U.S.P. 2,307,715, 5.1.43. Appl. 29.8.40. Hydrocarbons are dehydrogenated by contacting them at a high temperature with a catalyst consisting essentially of an intimate mixture of chromic sulphide and chromic oxide. The mixture is prepared by heating chromic sulphate in a reducing atmosphere to effect reduction of the sulphate to a mixture of the sulphide and oxide. H. B. M.

### Polymerization and Alkylation.

383. Patents on Polymerization and Alkylation. Anglo-Iranian Oil Co. E.P. 550,477, 11.1.43. Appl. 2.10.40. In a two-stage process for the production of high-octane-number gasoline, or a constituent thereof, from normally gaseous hydrocarbons boiling below 50° C., a hydrocarbon fraction, consisting of one or more alkanes, is subjected to isomerization at a pressure between 1 and 50 atmospheres and at a temperature between 250° and 600° C. Isomerization is carried out in the presence of a catalyst consisting of a metal oxide and/or a sulphide of Group 6 of the Periodic Table with the possible addition of metallic oxides or sulphides of Group 8. The *iso*-alkane produced is subjected, together with an alkene, to alkylation at moderate pressure and at a temperature between - 50° C. and + 100° C. in the presence of an alkylation catalyst. The alkylation product is distilled to yield liquid hydrocarbons having more than 6 carbon atoms.

E. T. Layng. U.S.P. 2,307,689, 5.1.43. Appl. 29.2.40. Olefinic hydrocarbons can be converted to higher-boiling hydrocarbons by contacting them at a high temperature with a catalytic contact material consisting of a plurality of porous granules of supporting material impregnated with the residue formed by heating a complex of ammonia and a metal pyrophosphate capable of reduction to a polymerization catalyst.

G. Egloff. U.S.P. 2,307,773, 12.1.43. Appl. 18.11.40. To convert paraffinic hydrocarbons into higher-molecular-weight paraffins the following procedure is adopted. A paraffin is fluorinated under conditions capable of producing a reaction mixture of alkyl fluoride and hydrogen fluoride. An *isoparaffin* is added to the mixture, which is then alkylated with the alkyl fluoride in the presence of hydrogen sulphide. H. B. M.

### Refining and Refinery Plant.

384. Patents on Refining and Refinery Plant. C. W. Tyson. U.S.P. 2,305,046, 15.12.42. Appl. 11.12.40. Treatment of a feed mixture for the separation of vaporized oil constituents from unvaporized oil constituents at temperatures at which the un-



vaporized constituents will further decompose. The feed mixture is passed into a cyclone separator at a temperature at which no vaporization of the liquid will occur. The walls of the separator are cooled by means of water circulated in a jacket round the walls. Vaporized constituents are withdrawn overhead and unvaporized constituents are withdrawn as bottoms.

D. I. Ashworth. U.S.P. 2,305,464, 15.12.42. Appl. 21.11.39. To remove impurities from oil which contains water a flowing stream of the oil-water mixture is established and then heated to a temperature designed to coalesce the impurities and render them readily separable from the oil. During heating the stream is maintained under pressure not lower than that required to prevent substantial vaporization of water. The stream is cooled to a temperature below the flash-point of the oil, and during the cooling process substantially the same pressure is maintained as that applied during heating. Afterwards pressure is substantially reduced without atomization and water plus impurities are separated from the oil.

W. W. Reed. U.S.P. 2,306,843, 29.12.42. Appl. 25.7.41. In a process for the caustic scrubbing of a petroleum distillate oil a partly spent aqueous caustic reagent is introduced into the vapours of the distillate oil, and flows concurrently therewith to and through condensation. Caustic reagent and condensate oil are separated to form a body of reagent through which condensate oil passes upwards and collects. Spent caustic reagent is removed, and collected partly treated condensate oil is passed upwards through the interstices of a packing material submerged in a body of caustic reagent for final treatment. Finished oil is collected and removed and fresh caustic reagent is supplied to the top of the caustic reagent body. Partly spent caustic reagent is removed from the bottom to supply the necessary partly spent caustic first introduced into the oil vapours.

R. E. Burk. U.S.P. 2,306,933, 29.12.42. Appl. 11.8.39. Sulphur compounds are removed from petroleum by eliminating hydrogen sulphide and afterwards heating the petroleum and separating it into lighter and heavier products. The heavier portion is conditioned for the action of metal carbonyl by subjecting it under mercaptan-forming conditions at a temperature of 400° F. to an agent for converting non-mercaptan sulphur into mercaptans. The treated material is subjected to the action of metal carbonyl at a temperature between 300° and 650° F. Finally all unreacted metal carbonyl is removed.

L. L. Lovell, W. River, P. E. Malson, and L. F. Bouillion. U.S.P. 2,306,993, 29.12.42. Appl. 30.6.41. A process for desulphurizing a normally liquid hydrocarbon distillate containing organic sulphur compounds. The distillate is contacted with a liquid slurry of an agent consisting essentially of clay in a hydrocarbon carrier oil at a temperature above that required for flash vaporization of the distillate and below that of incipient cracking under the conditions of the process. The slurry is heated to a temperature between 585° F. and the incipient cracking temperature. H<sub>2</sub>S is thus liberated, and distillate and H<sub>2</sub>S can be separated in vapour form from the slurry. The oil has a specific dispersion not above 115 and a resin content of less than 3%. It is free from asphalts and substantially non-volatile at the temperature of heating. The clay is previously activated by treatment with an electrolyte-yielding compound of an inorganic poly-oxy acid radical.

R. W. Henry and J. V. Montgomery. U.S.P. 2,307,130, 5.1.43. Appl. 25.3.40. To dewax mineral oils the waxy oil is first mixed with a dewaxing solvent. The mixture is chilled to precipitate wax, and the wax-oil-solvent compound is passed to a separating zone comprising washing and refluxing sections at a point intermediate between these two sections. A stream of chilled wash solvent is passed through this separating zone from the inlet end of the washing section to the outlet end of the refluxing section. In the stream of chilled wash solvent the oil and solvent from the precipitated wax-oil-solvent mixture are selectively dissolved. Into the oil-solvent mixture at a point adjacent to the outlet of the refluxing section an inert gas is passed. The inert gas completely dissolves in the mixture, and causes additional wax precipitation as reflux wax which flows by gravity in a direction countercurrent to the flow of the solvent-oil mixture and is washed by it. Reflux wax and precipitated wax are combined at the intermediate point of the washing and refluxing zones.

The combined waxes flow by gravity in a direction countercurrent to the flow of chilled wash solvent, and are washed with progressively purer wash solvent. Washed waxes are removed at the chilled solvent inlet end of the washing section. The solvent-dewaxed oil-dissolved gas mixture is removed at the solvent outlet end of the refluxing section.

R. L. Betts. U.S.P. 2,307,873, 12.1.43. Appl. 30.11.40. High-quality petroleum resins having softening points in the range 100°–200° F., with absolute colour below 1000, are segregated from extremely high-melting-point, dark-coloured resins having softening points above 200 in the following manner. A petroleum oil containing natural resins is treated with a phenolic solvent to form a raffinate phase and a homogeneous solvent-extract phase. Sufficient water is added to the extract phase to bring the ratio of the phenolic component in the solvent to water contained in the extract phase within the approximate limits 90 : 10 to 70 : 30. The extract phase is separated from substances which are not homogeneously contained therein, and the extract is freed of solvent. The solvent-free extract is distilled under sufficient vacuum to prevent substantial decomposition. Finally a high-quality resin distillate is separated.

F. A. Ferguson. U.S.P. 2,308,163, 12.1.43. Appl. 27.12.39. In a process for concentrating sulphuric acid containing carbonaceous material, a pool of acid is established in a stripping zone, and sulphuric acid and hot gases are fed concurrently into this pool, below the surface thereof, and in close proximity to each other. In this way incoming acid contacts the hottest gas first, and thus converts carbonaceous material in the acid into granular form.

H. B. M.

### Chemistry and Physics of Petroleum.

**385.\* Photomicroscopy of Salt in Petroleum.** L. F. Christianson and J. W. Horne. U.S. Dept. of the Interior. *Bur. Mines Technical Paper*, 638, 1942.—Until recently the composition of salts in or associated with petroleum has been determined by chemical methods, and the information gained in this way has been considered adequate for normal desalting operations. As pointed out in this report, however, the physical state and behaviour of the salts are important factors, and it is for this reason desirable that chemical analyses are supplemented with a knowledge of the physical form and mode of dispersion of these salts.

The report presents new data relative to the behaviour and type of emulsified brine droplets and to the natural physical structure and size of salt crystals found in petroleum. These data were obtained with the aid of a petrographic microscope, and observations were recorded and shown on photomicrographs. For the purposes of the investigation the physical state of salts of some of the oils being produced in the Mid-Continent area was studied. Initially, chemical analyses were made to determine the quantities of the predominating salts, and also to find the total quantity of water-soluble chlorides in the oils. Thereafter, photomicrographs were prepared to illustrate the variation in emulsions, the action of demulsifying agents, and the physical forms in which the salts crystallize from the demulsified oil.

It was found that the salt in the brine emulsion will ultimately precipitate as free or oil-coated microscopic crystals when the emulsion film has broken or when other conditions exist which permit the water to evaporate from the brine. It was concluded from these observations that it is better to make one complete dehydration of the emulsified oil at the time of treatment than to allow salt crystals opportunity to form from the partly demulsified brine.

Examination of various emulsions showed characteristic differences due to salt content, manner of producing the oil, size of droplets, and stability. Microscopical data revealed that the proper chemical destabilizer gave excellent results, but an improper chemical tended to create a very fine "tight" emulsion which might not be detected by the usual tests.

Careful study was made of the behaviour during refining of the salts in crude oils having a paraffin-intermediate base. Salts were examined and photographed in the crude oils before and after desalting, and in the refinery residual oils. Decomposition of the calcium and magnesium chlorides was found to be less in these oils than has been reported elsewhere.

H. B. M.



**386. Limits of Inflammability and Ignition Temperature of Ethyl Mercaptan in Air.** G. W. Jones, R. E. Kennedy, and W. E. Miller. U.S. Bur. Mines. *Report of Investigations*, No. 3648, June 1942.—In many types of fuel gases and dangerous atmospheres ethyl mercaptan ( $\text{CH}_3\text{CH}_2\text{SH}$ ) is used as an odorant to give warning of leakages of such gases into places where they might cause explosion or health hazards. By reason of its pungent smell, resembling that of onions, garlic, hydrogen sulphide, etc., ethyl mercaptan is particularly suitable for this purpose, and can be detected in very low concentrations. It is pointed out that because of its low boiling point ( $34.7^\circ\text{C}$ .) and correspondingly high vapour pressure at ordinary temperatures, ethyl mercaptan on exposure to air gives off enough inflammable vapour to create a decided explosion hazard. Experiments have been conducted to determine the limits of its inflammability in air and its ignition temperature in both air and oxygen. Results of these experiments are recorded in this report.

It was found that ethyl mercaptan becomes inflammable in dry air when the concentration reaches 28.0% by volume. During investigations of the upper limit of inflammability "cool-flame propagation" was observed. The flames were similar to those encountered in ether-air mixtures in the upper limit range. In a completely dark room, a long, very pale-blue flame was observed. On the basis of fourteen tests, in which the concentration of ethyl mercaptan ranged from 2.70% to 19.00% by volume, the upper limit of inflammability was established at  $18.2 \pm 0.2\%$  ethyl mercaptan, by volume, in air.

Minimum ignition temperatures of ethyl mercaptan were found to be  $299^\circ\text{C}$ . in air and  $261^\circ\text{C}$ . in oxygen.

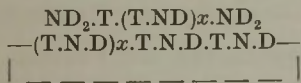
H. B. M.

**387. Separation of Gas Mixtures by Thermal Diffusion.** Anon. *J. Phys. Chem.*, 1942, 46 (7), 820.—On the basis of the kinetic theory, equations are given which enable the degree of separation to be calculated for a binary gas mixture under thermal diffusion conditions. These equations predict the effect of: the physical properties of the binary mixture components, the temperature gradient, the pressure, and the dimensions of the apparatus. To examine the validity of these equations a simple thermal diffusion apparatus has been constructed, and five gas mixtures containing hydrocarbons have been examined under various conditions. From the results of these experiments it is shown that the degree of separation obtained is less than that predicted from theoretical considerations, and that the effect of reducing the pressure is to increase the separation factor compared with a decrease predicted from theoretical considerations.

J. W. H.

**388. Patents on Chemistry and Physics of Petroleum.** M. de Groote. U.S.P. 2,305,067, 15.12.42. Appl. 25.6.41.—Process for resolving petroleum emulsions of the water-in-oil type which involves subjecting the emulsion to the action of a demulsifying agent consisting of a water-soluble ester.

M. de Groote. U.S.P. 2,306,329, 22.12.42. Appl. 27.6.40. To break a petroleum emulsion of the water-in-oil type it is subjected to the action of a demulsifying agent consisting of compounds of the kind selected from the class having the following type formulæ:



$x$  is a whole number less than 10, but including 0; T is an oxyhydrocarbon radical selected from the class consisting of the keto type, the ether type, and the alcohol type; D is selected from the class consisting of hydrogen, alkyl, alkylol, hydroxyalkoxyalkyl, aminoalkyl, hydroxyalkylamino, RCO, RCO.OX, RCO.OX', RCO.OX'', RCO.OX''', R'CO, R'CO.OX, R'CO.OX', R'CO.OX'' and R'CO.OX''' in which RCO is an acyl radical derived from a higher-molecular-weight carboxy acid having not more than 6 carbon atoms; R'CO is an acyl radical derived from a lower-molecular-weight carboxy acid having not more than 6 carbon atoms. OX represents a divalent radical obtained by removal of a hydroxyl hydrogen atom from a hydroxyalkoxyalkyl radical; OX' represents a divalent radical derived by the removal of an amino-

hydrogen atom from an aminoalkyl radical; and OX''' represents a radical derived by removal of a hydroxy hydrogen atom from a hydroxyalkylamino radical.

J. H. Hirsch. U.S.P. 2,306,606, 29.12.42. Appl. 21.3.39. In an apparatus for determining the rate of gas formation capable of directly reflecting the extent of cracking in a conversion apparatus there are incorporated means for withdrawing a sample of the products of conversion, means for cooling the withdrawn products to condense the normally liquid hydrocarbons, a separating chamber in communication with the cooling member and adapted to separate gases from liquid. Additionally there are devices for controlling the amount of cooled products introduced into the separating chamber, for regulating the cooling and maintaining a constant temperature in the separating chamber; for withdrawing liquid therefrom at constant rate. There is a gas outlet from the separating chamber and a device for measuring the amount of gas flowing in it.

R. M. Barrer. U.S.P. 2,306,610, 29.12.42. Appl. 31.1.42. In the separation of straight-chain hydrocarbons from mixtures containing these and at least one substance selected from the group consisting of branched-chain hydrocarbons and cyclic hydrocarbons, the mixture is contacted with crystalline zeolites having rigid three-dimensional atomic networks and having interstitial dimensions sufficiently large to sorb the straight-chain hydrocarbons, but sufficiently small to exclude other hydrocarbons.

C. M. Blair. U.S.P. 2,306,775, 29.12.42. Appl. 17.8.40. Petroleum emulsions of the water-in-oil type are broken by subjecting the emulsion to the action of a demulsifier consisting of a chemical compound selected from the class consisting of amines and substituted ammonium compounds in which a divalent radical, obtained by removal of a hydrogen atom directly linked to the carbon atom of an acyl radical, links a nitrogen atom to the acyl alcoholiform oxygen atom of a high molar hydroxylated carboxy-acid compound. The hydroxy-acid compound is derived from an acid the acyl radical of which contains not fewer than eight nor more than thirty-two carbon atoms. The remaining nitrogen valences are satisfied in any suitable manner by a member of the class consisting of hydrogen atoms, the same type of radical previously described, hydrocarbon radicals, hydroxy hydrocarbon radicals, acylated hydrocarbon radicals, ring structures in which the nitrogen atom is part of the ring, and anions.

M. de Groote and B. Keiser. U.S.P. 2,307,494, 5.1.43. Appl. 7.7.41. For breaking petroleum emulsions of the water-in-oil type, a demulsifier consisting of water-insoluble oxy-alkylated drastically oxidized castor oil is used.

M. de Groote and B. Keiser. U.S.P. 2,307,495, 5.1.43. Appl. 7.7.41. For breaking petroleum emulsions of the water-in-oil type a demulsifying agent consisting of a drastically oxidized water-insoluble oxy-alkylated triricinolein is used.

L. Liberthson. U.S.P. 2,307,744, 12.1.43. Appl. 17.5.41. Preparation of a composition capable of forming substantially stable, non-foaming aqueous emulsions of the oil-in-water type. The composition consists of a normally liquid petroleum hydrocarbon, an aqueous phase emulsifying agent of the water-soluble soap type, and 0.25-1.0% of chromium oleate calculated on the emulsifying base. H. B. M.

### Analysis and Testing.

389. **The Infra-Red Prism Spectrograph as a Precision Instrument.** R. A. Oetjen, C. L. Kao, and H. M. Randall. *Rev. Sci. Instr.*, 1942, 13 (12), 515.—The magnitude of the various factors which affect the accuracy of the infra-red spectrograph of the recording type has been investigated and the technique of the calibration of this instrument by means of the fine structure of CO<sub>2</sub>, NH<sub>3</sub>, and H<sub>2</sub>O is given. By the use of these compounds and a grating instrument of comparable resolution fifty absorption lines between 5  $\mu$  and 15  $\mu$  are obtained for making the empirical calibration.

J. W. H.

### Motor Fuels.

390. **Patents on Motor Fuels.** Anglo-Iranian Oil Co. E.P. 550,480, 11.1.43. Appl. 2.4.41.—Catalytic isomerization of normal paraffin hydrocarbons having at least four carbon atoms in the molecule is effected by contacting the original hydrocarbons



or a mixture containing normal paraffin hydrocarbons in the presence of hydrogen with a catalyst consisting of an oxide and/or a sulphide of a metal of Group 6 of the Periodic Table. Operating conditions are such that the normal paraffins may be isomerized or, alternatively, they may be converted in part to *isoparaffins* of lower molecular weight than the *isoparaffins* resulting from isomerization.

J. C. Munday. U.S.P. 2,305,026, 15.12.42. Appl. 7.12.38. To produce gasoline from low-boiling olefins and *isoparaffins*, the initial mixture is subjected to the action of an alkylation catalyst to produce a hydrocarbon having a wide boiling range. This product is divided into a heavy fraction and a light fraction. The latter fraction includes only those hydrocarbons which boil within the gasoline range. The heavy fraction is separately subjected to a further reaction with at least one hydrocarbon of the group consisting of *isoparaffins* and aromatic hydrocarbons. From the reaction mixture a hydrocarbon fraction is recovered which contains alkylated hydrocarbons boiling within the gasoline range. At least part of these are combined with the previously obtained light fraction.

D. A. Legge. U.S.P. 2,305,220, 15.12.42. Appl. 19.9.39. Hydrocarbon material, initially unsatisfactory for use as a motor fuel, is transformed into a high anti-knock gasoline by contacting the original material in vapour state and at a high temperature with a catalytic material consisting of an aluminium silicate. Silica and alumina are present in the catalytic material in the ratio of 0.8/1 to 1.2/1 by weight and the material is characterized by its fine porosity. It has a bulk specific gravity above 1.50 and a Mohs hardness above 2.5.

J. A. Chenicek. U.S.P. 2,305,673, 22.12.42. Appl. 29.2.40. To prevent depreciation of an olefin-containing hydrocarbon distillate, a gasoline gum inhibitor and a hydroxy alkylene diamine is added to it.

J. A. Chenicek. U.S.P. 2,305,674, 22.12.42. Appl. 29.2.40. To prevent depreciation of an olefin-containing hydrocarbon distillate, a gasoline gum inhibitor and a hydroxy-alkyl substituted-alkylene diamine is added to it.

J. A. Chenicek. U.S.P. 2,305,675, 22.12.42. Appl. 29.2.40. A gasoline gum inhibitor and an aryl substituted-alkylene diamine are added to an olefin-containing hydrocarbon distillate to prevent deterioration.

J. A. Chenicek. U.S.P. 2,305,676, 22.12.42. Appl. 29.2.40. A gasoline gum inhibitor and a substituted alkylene diamine, wherein the substituent group is an amino-alkyl group, are added to cracked gasoline to prevent deterioration.

W. K. Simpson and C. G. Dryer. U.S.P. 2,305,742, 22.12.42. Appl. 25.3.39. In the treatment of gasoline with solid adsorbing refining agents, the life of the adsorbent agent is prolonged by treating the gasoline in the first place with a relatively small amount of a dilute aqueous solution of a mineral acid. The quantity of the solution is insufficient to effect any substantial refining of the gasoline. Afterwards the gasoline is refined by contacting it with the solid adsorbent at a temperature between 200° and 600° F.

F. M. McMillan. U.S.P. 2,306,253, 22.12.42. Appl. 29.11.41. The following processes are incorporated in the production of branched-chain hydrocarbons in the absence of any substantial hydrocarbon decomposition. A substantially paraffinic hydrocarbon mixture containing C<sub>4</sub> and C<sub>5</sub> hydrocarbons is fractionated in a fractionating zone to separate fractions respectively rich in normal butane and normal pentane. The normal butane fraction is contacted at a temperature between 100° and 200° C. with an aluminium halide catalyst in a first isomerizing zone. The pentane fraction in admixture with the effluent from the first isomerizing zone is contacted at a temperature between 40° and 120° C. with an aluminium halide catalyst in a second isomerizing zone. *iso*Butane and *isopentane* are separated from the effluence of the second isomerizing zone and, in admixture with a fraction predominating in butylene and butane, they are contacted under alkylating conditions with an alkylation catalyst. In this way *isobutane* and *isopentane* are reacted with butylene. Butane, *isobutane*, and *isopentane* are separated as a vapour fraction from the products of alkylation, and this vapour fraction is passed to the fractionating zone.

H. E. Drennan and J. L. Farrell. U.S.P. 2,308,063, 12.1.43. Appl. 12.10.39. To improve the anti-knock qualities of liquid hydrocarbons, they are first vaporized and

the vapours are superheated to a temperature between 600° and 1500° F. Superheated vapours are passed through a bed of catalytic material of the clay type for the improvement of octane number. The catalytic material has previously been spent in the treatment of hydrocarbons to improve colour and gum characteristics. The quantity of catalytic material is so regulated in each stage that it is spent at approximately the same time for the specific improvement which it imparts to the hydrocarbon gases.

H. B. M.

### Lubricants and Lubrication.

**391.\* Conservation of Lubricating Oil.** Oil Advisory Committee. Transvaal Chamber of Mines. *J. Inst. Pet.*, December 1942, 28 (228), 285-296.—A practical summary of suggestions made towards conserving lubricants on storage and use is presented. The report deals with lubricants for air-compressor cylinders, steam-engine cylinders, locomotives, wick oilers, bottle oilers, rock-drills, air motors, air-operated pumps and drill sharpeners, greases for plain bearings, ball and roller bearings, cocopan axles and open gears.

Further, the report treats briefly the problems met in compressor crank-cases, steam-engine crank-cases, steam-turbines, electrically-driven turbo compressors, hoist hydraulic systems, gear-boxes, gyratory crushers, diesel locomotives, ring-oiled bearings, motor-transport transformer and switch oils, cutting oils and compounds, and rope dressings for wire ropes.

Reconditioning of oils is outlined.

A. H. N.

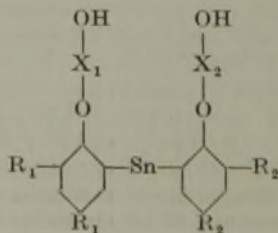
**392. Patents on Lubricants and Lubrication.** S. J. M. Auld, E. G. Ellis, and H. A. Walker. E.P. 550,429, 7.1.43. Appl. 25.7.41.—In the manufacture of lubricating grease from mineral oil and soaps, the component materials are passed at suitable temperatures to separate containers. The contents of the containers are delivered in fixed proportions by separate mechanical devices to a mixer large enough to accommodate the individual supplies and to allow reaction and homogenizing to be completed. It is claimed that the process renders unnecessary costly and complicated pans and kettles, stirring mechanism, etc.; reduces heating costs as the greases are made at low temperature; and decreases power consumption.

E. R. Barnard. U.S.P. 2,304,874, 15.12.42. Appl. 26.9.40. An improved lubricant is prepared from a narrow-cut solvent-extracted petroleum oil having a 90% distillation under 1 millimetre mercury pressure not substantially greater than 200° F. above the initial boiling point, and from 1% to 40% of an *isobutylene* polymer having a Saybolt viscosity at 210° F. of 185-500 sec. and a flash-point between 350° and 450° F.

R. Rosen and B. W. Geddes. U.S.P. 2,305,034, 15.12.42. Appl. 20.12.39. An improved mineral-oil composition is prepared by admixing with a petroleum-oil fraction not more than 5% of an organic base having a heterocyclic structure containing nitrogen, and not more than 0.1% of sulphur. The sulphur is present in an organic compound which is an antioxidant for petroleum oils.

B. H. Lincoln and G. D. Byrkit. U.S.P. 2,305,627, 22.12.42. Appl. 24.5.39. A lubricant is prepared by adding a small proportion of a tin- and halogen-bearing reaction product of a stannic halide and a beta-diketone to an oil of lubricating viscosity.

E. W. Cook and W. D. Thomas, Jr. U.S.P. 2,306,354, 22.12.42. Appl. 29.11.41. Preparation of a lubricant containing a compound of the general formula:





$X_1$  and  $X_2$  are alkylene radicals containing two to four carbon atoms inclusive,  $n$  is a positive integer not more than two, and  $R_1$  and  $R_2$  are members of the group consisting of alkyl, *cycloalkyl*, and hydrogen radicals. At least one  $R_1$  and at least one  $R_2$  are members of the group consisting of alkyl and *cycloalkyl* radicals.

E. B. McConnell and J. M. Musselman. U.S.P. 2,306,971, 29.12.42. Appl. 16.8.39. A small amount of the reaction product obtained by heating molar proportions of triamylamine and sulphur is added to lubricating oils to improve their quality.

J. C. Zimmer and A. J. Morway. U.S.P. 2,307,183, 5.1.43. Appl. 9.5.39. An extreme-pressure lubricant consists of a mineral lubricating oil, an oil sulphurized fatty oil, and a soluble reaction product of phosphorus sesquisulphide with a halogenated aliphatic hydrocarbon compound containing at least 5% halogen.

B. H. Shoemaker. U.S.P. 2,307,307, 5.1.43. Appl. 12.9.38. Preparation of an extreme-pressure lubricant containing a lubricating oil and a thioketo polysulphide having the general formula  $(RXC = S)_2S_n$ .  $R$  is a radical selected from the group consisting of alkyl, chloralkyl, aryl, and chloraryl radicals;  $X$  is an element selected from the group consisting of oxygen and sulphur; and  $n$  is an integer greater than two. The thioketo polysulphide is used in small but sufficient quantities to impart extreme pressure qualities to the lubricant.

E. J. Barth. U.S.P. 2,307,615, 5.1.43. Appl. 8.3.41. A liquid lubricating-oil composition is prepared by admixing with a petroleum lubricating oil a small amount of an oil-soluble calcium phenolate selected from the group consisting of calcium ortho- and para-phenolates and substituted derivatives thereof having at least one alkyl group directly attached to the basic phenol ring.

A. H. Gleason. U.S.P. 2,307,885, 12.1.43. Appl. 16.7.40. Preparation of a lubricating product consisting of a hydrocarbon lubricating oil blended with 0.2-5% of a vinyl phenol polymer in which the hydroxyl groups are esterified to render the polymer soluble in the hydrocarbon oil.

I. Silverman. U.S.P. 2,308,116, 12.1.43. Appl. 11.7.40. Preparation of a lubricant of relatively low, substantially non-fugitive, non-reverting pour point consisting of a wax-containing petroleum hydrocarbon oil, having normally a relatively high pour point due to the wax, a metallic soap pour point depressant, and an oil-soluble metallic petroleum sulfonate.

T. G. Rochner and L. H. Sudholz. U.S.P. 2,308,427, 12.1.43. Appl. 16.10.40. Sulphurized Vinifera palm oil is added to a mineral-oil lubricant to confer enhanced extreme-pressure lubrication qualities. If desired the Vinifera palm oil can previously be reacted with phosphorus.  
H. B. M.

## Asphalt and Bitumen.

393. Patents on Asphalt and Bitumen. B. A. Anderton. U.S.P. 2,304,773, 15.12.42. Appl. 16.7.38. A heat-stable bituminous product, especially suitable for application by remelting, is obtained in the following manner. A digesting medium consisting of tar-oil and pitch constituents is heated with between 7% and 25% of a coal of the group consisting of bituminous and sub-bituminous coal to a temperature between 20° and 100° above the digestion temperature corresponding to a product of maximum softening point. The final bituminous product has a softening point between 50° and 90° C., a penetration at 0° C. of at least four and a penetration at 25° C. of not more than five times the penetration at 0° C.

H. M. Noel. U.S.P. 2,305,440, 15.12.42. Appl. 25.7.40. A hard, unoxidized bituminous material suitable for use in a storage-battery compound is prepared from cracked and vacuum distilled paraffin-free asphaltic-base petroleum crude oil having a susceptibility factor between 27 and 35, and a softening point between 320° and 420° F. Material insoluble in carbon disulphide is less than 1.5%. Manufacture entails cracking a paraffin-free asphaltic-base West Venezuela crude to a residual bottoms

oil having a gravity viscosity relationship of 8-9° A.P.I. gravity and Furol viscosity of between 1000 and 1400 sec. to between 600 and 850 sec. at 122° F. respectively. The residual oil is distilled under vacuum to obtain a residue of bituminous material of desired viscosity and susceptibility factor.

R. L. Ortynsky. U.S.P. 2,308,245, 12.1.43. Appl. 28.6.41. To produce a Mexican-type paving composition an asphalt of the following composition is oxidized: Asphaltenes 1.5-5%; petroleum resins, 55-65%; and aromatic oil the balance. The resins and the oil have specific dispersions above 200 and flash-points above 500° F. The oxidized asphalt has an asphaltene content of 50-60% and a softening point of at least 360° F. This is fluxed with 1-1-1.6 parts of petroleum extract per part of oxidized asphalt. The extract has a Saybolt furol viscosity of 30-200 at 275° F. and a flash-point of at least 500° F. With the resulting fluxed asphalt is blended an amount of a hydrocarbon distillate sufficient to produce a blended asphalt having a penetration of 25-200 at 77° F.

H. B. M.

### Special Products.

**394.\* Chemicals from Petroleum—Formaldehyde.** W. T. Ziegenhain. *Oil Gas J.*, 15.10.42, **41** (23), 34.—Substantial quantities of formaldehyde are being made by controlled oxidation of natural gas, the largest plant being located in the Mid-Continent.

The principal commercial products obtained are formaldehyde, methanol, and acetaldehyde. Methods of manufacture and separation of the three products from one another are briefly described and properties and applications of formaldehyde are outlined. Production from natural gas has been on a continuous and expanding basis since 1928.

R. A. E.

**395. Progress in Plant Insecticides.** S. H. Harper. *Chem. Prod.*, November/December 1942, **6** (1/2) 5.—A review, with bibliography, is given of recent developments in the use, structure, and determination of vegetable insecticides—*e.g.*, nicotine, pyrethrum, derris and cubé roots, and quassia. The problem of poisoning of honey-bees by lead arsenate sprays is being attacked by the testing of various essential oils as repellents, and of nicotine-bentonite oil-sprays, which are claimed to be as toxic to pests but less phytocidal. The export of concentrated extracts of pyrethrum, instead of the flowers, and cultivation in this country is suggested. Pyrethrum extracts in kerosine are recommended for control of bugs, etc., in air-raid shelters, and mists of pyrethrum in mineral oil for control of pests in stored food produce. In oil solution there is little difference between the toxicities of pyrethrum I and pyrethrum II, although the latter is chiefly responsible for knockdown. More attention is being paid to the chemical and biological standardization of pyrethrum. The risk of dermatitis when pyrethrum preparations are used for scabies and louse control is believed to be due to the presence of a steam volatile essential oil. Pyrethrum is also effective as a repellent for midges and mosquitoes. Reduction of the toxic concentration of pyrethrum is being attempted by the use of synergists such as sesame oil.

The structure and toxicity of the constituents of derris and cubé roots are being investigated, it being shown that rotenone is not the only active constituent. Previous constituents extracted by alkali and found to be ineffective are now believed to have been present in the optically active form, but have suffered racemization by alkali and become non-toxic. A method of evaluating the toxicity from the sum of the rotenone concentrate,  $\frac{1}{8}$ th of deguelin concentrate and  $\frac{1}{5}$ th of the toxicarol concentrate has now been worked out. Derris solutions in petroleum oil sprays, containing a mutual solvent, are giving better control of citrus-red scale in California. Control of head louse, body louse, and scabies by rotenone or derris-root creams, lotions, etc., is reported. Quassia is reported as giving good control of sawfly.

C. L. G.

**396. Acid Sludge in the Manufacture of Superphosphate.** Anon. *Chem. Trade J.*, 19.2.43, **112**, 166.—Acid sludge from the Haifa refinery, containing about 50%  $H_2SO_4$ , is being supplied to manufacturers of superphosphate for fertilizer production. A



good quality superphosphate can be made with this acid tar from Transjordan phosphate (69-75% tricalcium phosphate).  
C. L. G.

**397. Patents on Special Products.** Anglo-Iranian Oil Co. E.P. 550,389, 6.1.43. Appl. 5.3.41.—The patent relates to a catalytic process for the preparation of 2:4-dimethylpentane from 2:3-dimethylpentane or of 2:3-dimethylpentane from 2:4-dimethylpentane. The hydrocarbon which is to be converted is contacted with concentrated sulphuric acid as catalyst under conditions of vigorous stirring of the reaction mixture at a temperature in the range  $-10^{\circ}\text{C}$ . to  $50^{\circ}\text{C}$ . In addition to the advantages to be derived from increased octane number by converting 2:4-dimethylpentane to the 2:3 form, the reverse conversion is advantageous, for instance, when the 2:3 isomer is associated with low-octane-number paraffins, of nearly related boiling point, such as methylhexanes, from which it cannot be separated by fractionation.

J. G. Fife. E.P. 550,776, 25.1.43. Appl. 3.6.41. In a process for the treatment of saturated non-cyclic hydrocarbons in the vapour phase with an aluminium halide in the presence of a hydrogen halide, the concentration of the hydrogen halide in the treating zone is maintained below that causing degradation of the hydrocarbons under the conditions of treatment. As the catalyst declines in activity the concentration of the hydrogen halide in the treating zone is increased. The process has particular application to the vapour-phase isomerization of butane with the aid of aluminium chloride.

F. O. Rice and W. D. Walters. U.S.P. 2,305,635, 22.12.42. Appl. 25.10.40. Higher-molecular-weight hydrocarbons are converted to lower-molecular-weight hydrocarbons by heating to a conversion temperature in the presence of a relatively small amount of diacetyl.

S. H. White. U.S.P. 2,305,752, 22.12.43. Appl. 2.10.40. A gaseous fuel suitable for use with oxygen in a blow-pipe contains a gas of low calorific value from the group consisting of coal gas, natural gas, and illuminating gas, mixed with propane and ether. Proportions are approximately 1 part of propane to 0.375 part of ether. The quantity of propane and ether is sufficient to increase the calorific value of the gas, and in any event is not materially less than 20%.

S. H. White. U.S.P. 2,305,753, 22.12.42. Appl. 2.10.40. A gaseous fuel suitable for use with oxygen in a blow-pipe contains a member of the group consisting of propane and butane mixed in a liquid stated with not less than 16% nor more than 21% by weight of ether. The fuel is used in the same way as acetylene or hydrogen in application to metals.

E. M. Marks. U.S.P. 2,306,218, 22.12.42. Appl. 27.9.41. Hydrocarbon stock is converted into other hydrocarbons by contacting it with a catalyst consisting of aluminium oxide and an alkali metal borate at suitable temperature. The catalyst contains 5-15% by weight of water.

L. Liberthson and M. Blumer. U.S.P. 2,307,743, 12.1.43. Appl. 7.1.41. To obtain an improved petroleum sulphonate product a solution of a petroleum sulphonate stock, derived from the acid treatment of a petroleum lubricating-oil distillate under sulphonating conditions, is contacted in the presence of water, in a solvent for the sulphonate with an alkali metal chlorite. Conditions are adjusted in regard to quantities and length of contact to produce a product having a Lovibond colour of less than 8R at a pH substantially below 7. Afterwards a petroleum sulphonate product substantially stable during storage and substantially non-darkening at elevated temperatures is recovered.  
H. B. M.

## Detonation and Engines.

**398. Patent on Detonation and Engines.** G. B. Banks. U.S.P. 2,306,372, 29.12.42. Appl. 26.3.40.—A gasoline knock-recorder comprises a tubular member connected at one end to a source of variable fluid-pressure supply, primary and secondary transformer coils about a portion of that member, a core which slides in the member and which

is normally held substantially within the magnetic field of the primary coil. Movement of the core in the direction of the secondary coil transfers electric energy from the primary to the secondary coil. A reduced-diameter bypass duct is connected to the tubular member to bypass a quantity of fluid pressure to the end of the core adjacent to the secondary coil. The movement of the core is thus cushioned in one direction.

H. B. M.





# INSTITUTE NOTES.

APRIL, 1943.

## CANDIDATES FOR ADMISSION.

The following have applied for admission to the Institute. In accordance with the By-laws, the proposals will not be considered until the lapse of at least one month after the publication of this *Journal*, during which time 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 the candidate.

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

The names of candidates' proposers and seconders are given in parentheses.

GOSSLING, Percy William L., Research Chemist, W. B. Dick & Co., Ltd.  
(*A. J. Sear; A. C. Michie.*)

MOORE, Harold, Consulting Technologist. (*Dr. A. E. Dunstan; C. Dalley.*)  
(Application for transfer from Member to Fellow.)

HALLAM, Thomas A., Research Chemist, Shell Refining and Marketing Co., Ltd. (*P. M. Griffith; G. D. Hall.*)

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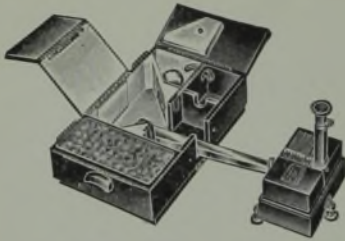
## FORTHCOMING MEETINGS.

Meetings have been arranged for the 29th April, 27th May, and the 24th June, and will be held at the Royal Society of Arts, John Adam Street, Adelphi, W.C.2, on those days at 5.0 p.m.

On the 29th April, Professor V. C. Illing will give a paper on "**Education and the Petroleum Industry.**"

ARTHUR W. EASTLAKE,  
ASHLEY CARTER,  
*Joint Honorary Secretaries.*

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- CHART D: Kinematic Viscosity, Low Range (20 by 20 in.)—temperature range, — 30° F. to + 450° F.; viscosity range, 0.4 to 100 centistokes. Price 7s. 6d. per pad of 25.

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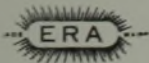
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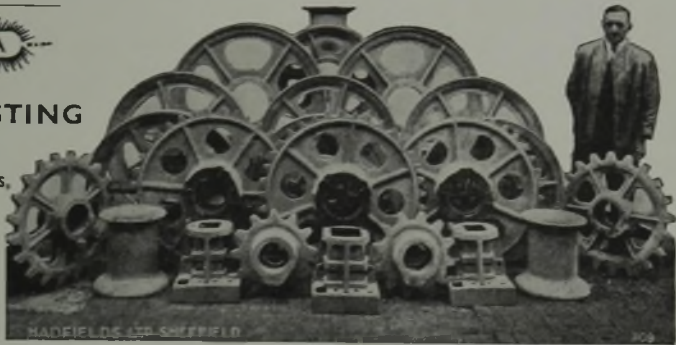
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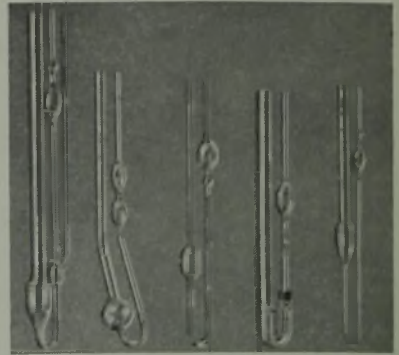
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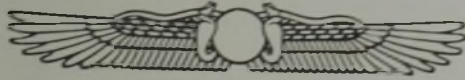
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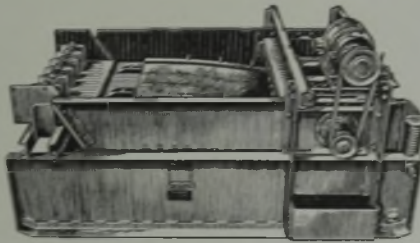
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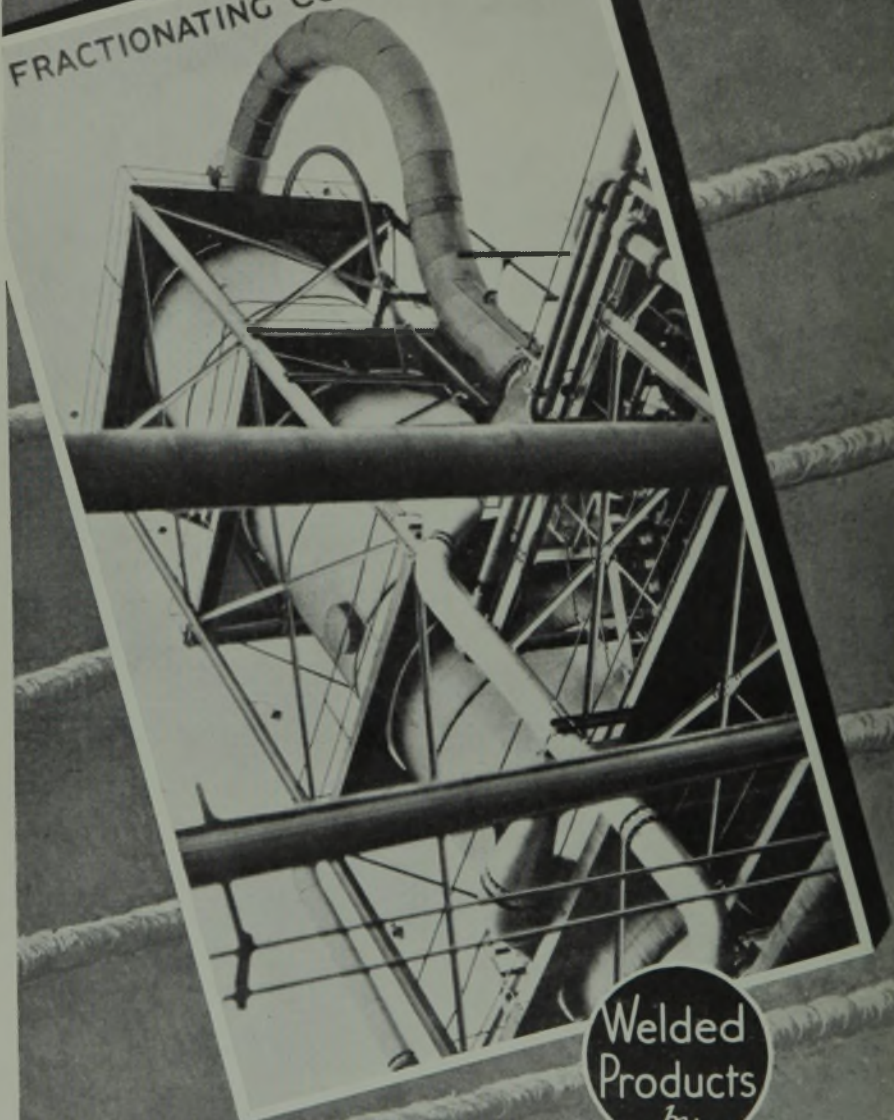
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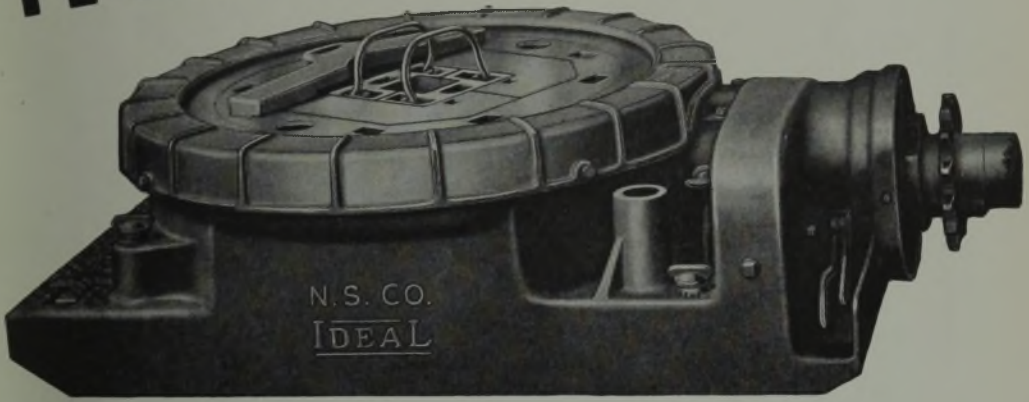
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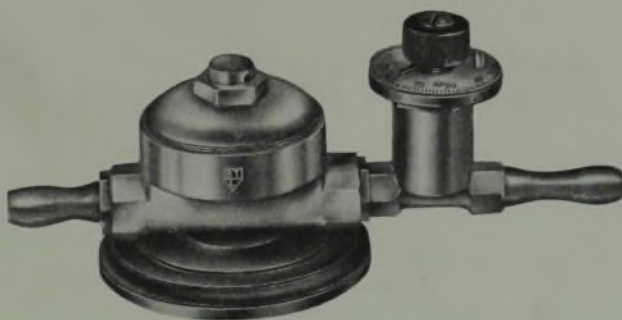
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# COMBINED GOVERNOR AND FINE ADJUSTMENT VALVE

*for experimental fractionation  
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This combined governor and fine adjustment valve provides micrometer adjustment on a stabilised gas pressure. It is invaluable for running **fractionations** and ensuring **reproducible results from routine range tests**.

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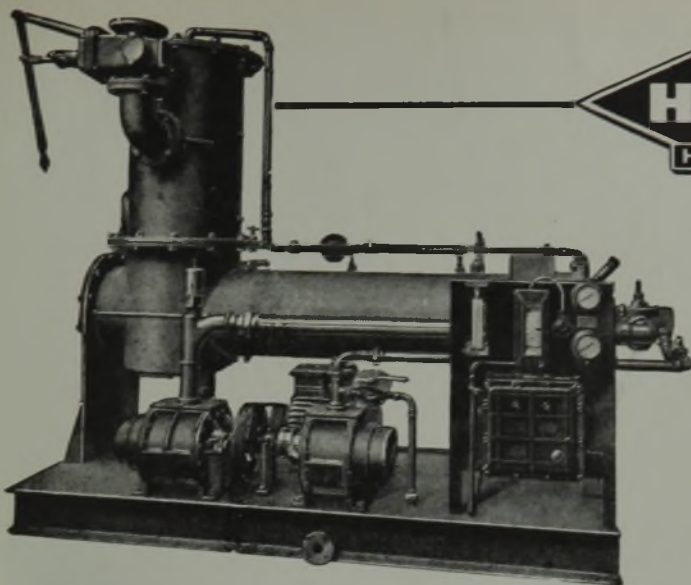
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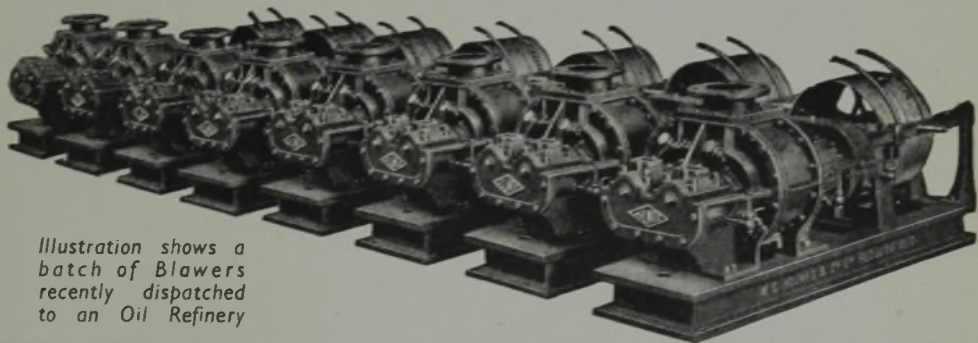
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35/37% (Alumina)



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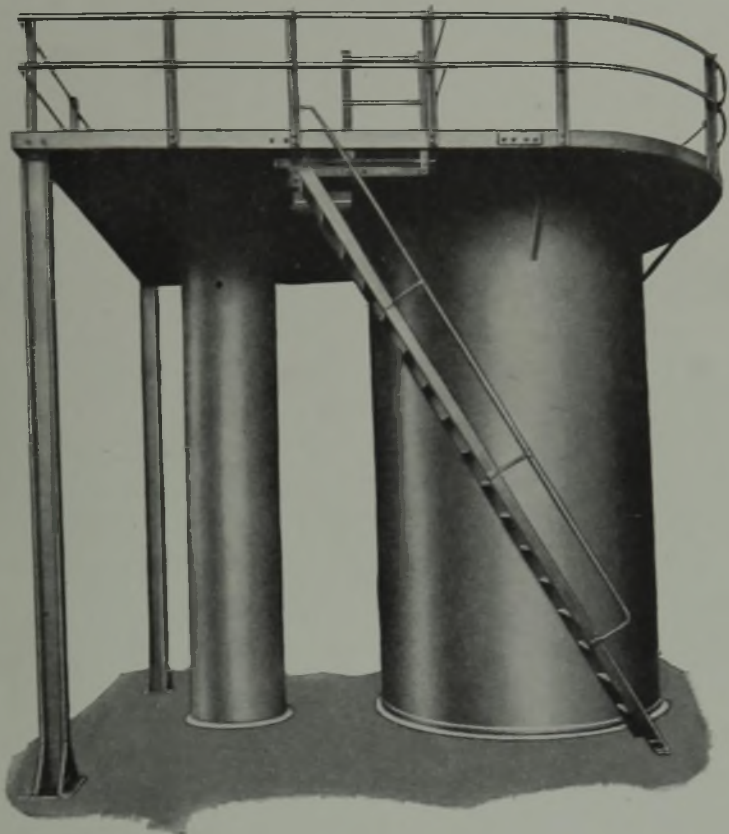
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