

PROCESS DEVELOPMENT AND PRODUCTION OF ISOHEXANE AND ISOHEPTANE AS AVIATION FUEL COMPONENTS.

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SUMMARY.

Early in 1940 the main components of 100 octane fuel were either hydrocarbon fractions isolated from crude by physical means, such as distillation or solvent extraction, or, alternatively, were hydrocarbon fractions produced by synthetic processes, such as pyrolysis, alkylation, and catalytic polymerization followed by hydrogenation. Components such as these were then blended with a suitable base stock and T.E.L. to give the final 100-octane fuel, and it was due to the difficulty experienced in the production of a suitable base stock that a programme of work was instituted which culminated in 1944 in the production of 315,000 gallons per day of refined isohexane and isoheptane fractions, representing about 35 per cent. of the daily output of 100-octane aviation fuel from the Anglo-Iranian Oil Company Refinery at Abadan. The output of these two components is obtained from eight fractionating units, each 12 feet in diameter, four being fitted with 50 bubble-trays, two with 76, and two with 100 bubble trays. For constructional reasons the 76- and 100-plate units were built as double columns, so that there are twelve columns in all, varying between 120 and 140 feet in height. The complete superfractionation installation, including staging, exchangers, pumps, and pipework, represents 6300 short tons of steel, which is equivalent of 0.7 ton steel per barrel per day of useful product.

PART I.—DEVELOPMENT OF PROCESS.

INTRODUCTION.

THE outbreak of war in 1939 naturally brought attention to bear on the manufacture of 100-octane number aviation spirit in increasing quantities. The Air Ministry Specification which had to be met at that time is given below, and in addition it was necessary that the spirit should have a rich mixture rating which equalled or exceeded the Standard A.M. 100 Reference Fuel, when tested in the Pegasus single-cylinder engine.†

S.G. at 60° F.	0.790 max.
Dist. to 75° C.	10% min.
" 100° C.	50% min.
" 150° C.	90% min.
F.B.P.	180° C. max.
Corrosion test (I.P.T. G4b)	1 mgm./100 ml. max.
Vapour pressure	7 lb. max.
Gum existent	10 mgm./100 ml. max.
" potential	10 mgm./100 ml. max.
Freezing point	-60° C. max.
T.E.L. content	4.0 ml./I.G. max.
Octane No. (C.F.R.M.)	100

* Anglo-Iranian Oil Company.

† The whole of the research and development work described in this paper was carried out using the Pegasus engine for rich mixture tests. Pegasus ratings have, however, been converted to the equivalent F4 (3-C) ratings throughout, in order to bring them into line with recent practice.

A considerable amount of knowledge existed concerning the factors which controlled the various points of this specification, but not so much was known about those affecting rich mixture performance. A source of supply of high octane number material was available in *iso*-octane, hydrocodimer, and alkylate, and it was possible to visualize a considerable increase in this, particularly from the alkylation process. The first step to be taken was to find a suitable base-stock to blend with this. Cracked products were not permissible, and it was soon evident that while the use of conventionally produced straight-run blend stocks from Iranian crude would permit blends to be made meeting the above specification, these blends would be seriously low in rich mixture performance. If such base spirits were used, the aviation spirits produced would have to have had octane numbers well above 100 if they were to meet the rich-mixture requirement.

Intensive work was carried out in order to unravel the factors affecting rich-mixture performance, and to establish which of the various hydrocarbon types was good in that respect. The conclusion was soon reached that the material boiling above 100° C. would have to be predominantly aromatic in character. The production of such an aromatic concentrate, containing toluene and xylenes, was not difficult in principle. Although the concentrations of aromatics in Iranian crude are not high, they are such that a large amount of aromatic-rich blend stock could be produced by preparing a suitable cut on the crude distillation units and submitting it to solvent extraction. As a result, it was possible to make accurate estimates of the recoverable yield and quality of this material from the total crude being processed in the refinery, and to prepare representative samples at the Research Station for both quality assessment and the production of trial blends. In other words, it was possible to fix one of the components of the final aviation spirit blend both as regards quality and quantity.

A second component which could also be assessed in the same way was isopentane. The amount present in the crude was accurately known, and plant was already installed in Abadan for its production, so that the recoverable yield could be determined by actual refinery operation.

A third component, as has already been mentioned, was the *iso*-octanes in their various forms. Some production capacity existed, and it could be seen that more would probably have to be provided. Since, however, the production of this key component would be expensive, and might be the bottle-neck in the overall quantity of 100 octane-number spirit producible, it was very desirable to keep the percentage in the final blend as low as possible.

The establishment of the various facts just given allowed attention to be focussed more intensively on the remaining material which was available and which could usefully be included in the final blend. Assuming that this material, which would be in the nature of a base-stock, would have to be obtained from the straight-run spirit, the possibilities were confined to the hydrocarbons boiling in crude in the range 30–100° C. approximately. It is with the production of suitable material boiling in this range, of such quality that a reasonably large proportion could be blended in with the other components enumerated above, that this paper is concerned.

BASIC CONSIDERATIONS.

The first step was to examine such information as was available on the octane ratings of the lower-boiling paraffins and naphthenes. These are tabulated, together with the boiling points of the individual hydrocarbons, in Table I.

TABLE I.
Boiling Points and Octane Numbers of Low-boiling-range Hydrocarbons.

	Hydrocarbon.	Boiling point, ° C.	Octane number C.F.R. MM.
	2 : 2-Dimethylpropane (<i>neopentane</i>)	9.45	83
	2-Methylbutane (<i>isopentane</i>)	27.95	90
	<i>n</i> -Pentane	35.95	62
	<i>cyclo</i> Pentane	49.4	85
	2 : 2-Dimethylbutane (<i>neohexane</i>)	49.7	93
	2 : 3-Dimethylbutane (<i>diisopropyl</i>)	58.0	94
	2-Methylpentane	60.3	73
	3-Methylpentane	63.3	75
	<i>n</i> -Hexane	68.8	26
	Benzene *	80.2	1.0 + 2.75 c.c. T.E.L./U.S.G.†
	Methylcyclopentane	71.85	80
	2 : 2-Dimethylpentane	78.9	93
	2 : 4-Dimethylpentane	80.65	82
	<i>cyclo</i> Hexane	80.8	77
	2 : 2 : 3-Trimethylbutane (<i>triptane</i>)	80.9	1.0 + 0.07 c.c. T.E.L./U.S.G.‡
	3 : 3-Dimethylpentane	86.1	84
	1 : 1-Dimethylcyclopentane	88.05	(80)
	2-Methylhexane	89.7	(45)
	2 : 3-Dimethylpentane	89.9	89
	1 : 3-Dimethylcyclopentane (<i>trans</i>)	90.5	(80)
	3-Methylhexane	91.9	(45)
	1 : 2-Dimethylcyclopentane (<i>trans</i>)	91.9	(80)
	<i>n</i> -Heptane	98.4	0

() = estimated values.

* Benzene is located in this position due to the abnormal vapour-liquid relationship existing between it and *n*-hexane, as discussed by Tongberg and Johnstone (*Ind. Eng. Chem.*, 1933, 25, 733-5).

† Blending octane number 91.

‡ Blending octane number 113.

It will be observed that all the hydrocarbons boiling in the range 49.4-63.3° C. have octane numbers equal to or greater than 73. In consequence, a cut on any crude boiling between these temperature limits must possess, provided *n*-pentane and *n*-hexane are completely removed, an octane rating equal to or greater than 73, the exact value depending on its actual composition. For the production of such a cut commercially it is necessary to separate between *n*-pentane (b.pt. 35.95° C.) and *cyclopentane*, (b.pt. 49.4° C.) at the front end, and between 3-methylpentane (b.pt. 63.3° C.) and *n*-hexane (b.pt. 68.8° C.) at the back end. Furthermore, it is evident that the quality of such a cut will be dependent on the amount of *cyclopentane* (octane number 85) and *neohexane* (octane number 93) present, and a high recovery of these components must be aimed at in the fractionation process.

Similarly, it would appear probable from a further inspection of Table I that a cut taken between 70° C. and 92° C. would also have considerable value as an aviation component. Such a cut covers the range of hydrocarbons including benzene, methylcyclopentane, cyclohexane, the isohexanes, and the dimethylcyclopentanes, but, due to the presence of

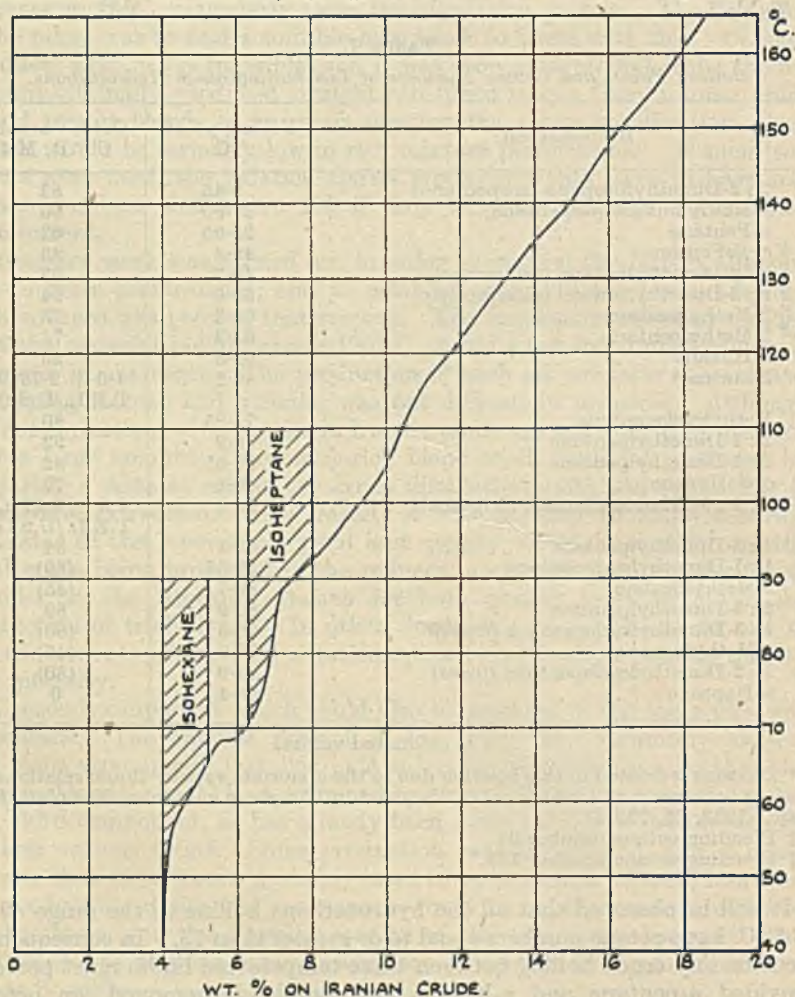


FIG. 1.

TRUE BOILING-POINT DISTILLATION ON TYPICAL IRANIAN CRUDE.

2- and 3-methylhexanes, no useful forecast can fairly be made of the octane rating of such a cut unbacked by experimental work. Its production is dependent on the separation between *n*-hexane (b.pt. 68.8° C.) and benzene-methylcyclopentane (b.pt. about 70° C.) at the front end; and between 3-methylhexane-1 : 2-dimethylcyclopentane (b.pt. 92° C.) and *n*-heptane (b.pt. 98.4° C.) at the back end.

It should be pointed out that at this stage we had no knowledge as to the probable behaviour of these cuts as regards rich mixture performance.

ASSESSMENT OF CRUDE: SEMI-SCALE PRODUCTION.

Having established the various hydrocarbons whose presence was likely to be acceptable in the proposed base stock, it was next necessary to examine the available crude and determine the quantities present. We

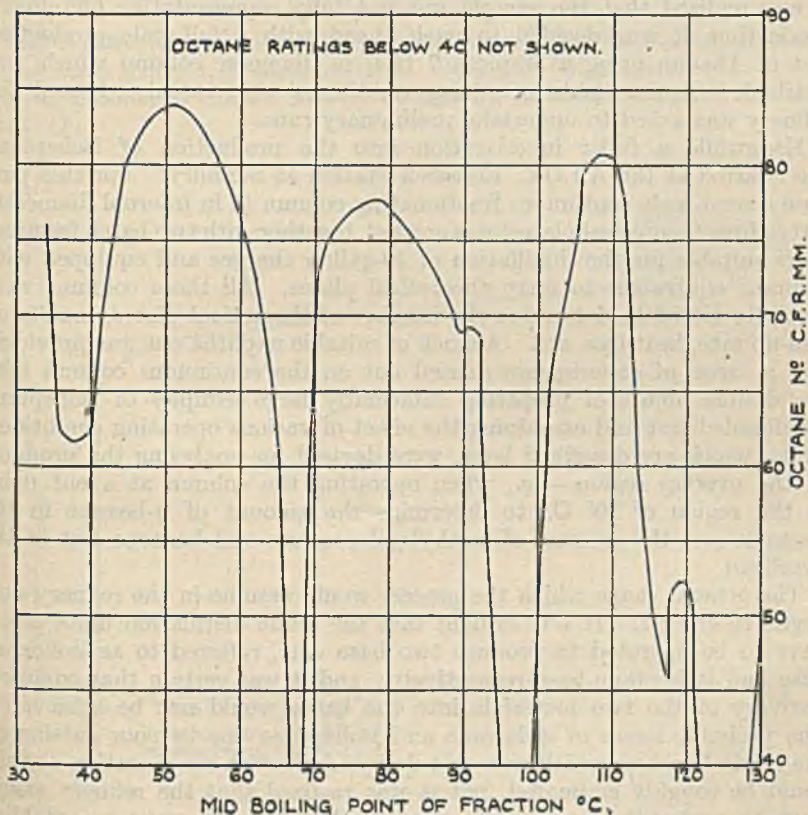


FIG. 2.

OCTANE NUMBER—BOILING POINT RELATIONSHIP FOR TYPICAL IRANIAN CRUDE.

were fortunate at this stage in having at our disposal the results of a careful fractionation of a typical Iranian naphtha which had been carried out by Professor Fenske, at Penna. State College, using a 40-gal. charge and a column equivalent to seventy-five theoretical plates operated at high reflux ratio. The cuts produced in this fractionation had been sent to Sunbury for detailed examination, and the data which were obtained from this are shown in Figs. 1 and 2. In Fig. 1 the approximate limits of the isohexane and isoheptane cuts as defined in Table I are indicated showing that approximately 1.2 per cent. by weight of isohexane, and

1.7 per cent. by weight of isoheptane should be available, and if the quality was acceptable it was evident that these materials would make a most valuable contribution to the final blend.

The next steps were concentrated on the production of isohexane, there being less doubt that the quality of this cut would be acceptable. A quantity of 2 gallons was prepared by fractionating Iranian naphtha batchwise in a 25-plate column, and the octane number and Pegasus blending number determined. The results were encouraging, and although it was realized that the sample was not fully representative of refinery production, it was decided to push ahead with a full-scale production test in Abadan using a 50-plate 7 ft 6 in diameter column which was available. Approximate operating conditions were calculated, and the refinery was asked to undertake preliminary runs.

Meanwhile a fuller investigation into the production of isoheptane was started at the A.I.O.C. Research Station at Sunbury. For this purpose a semi-scale continuous fractionating column (4 in internal diameter; forty-three theoretical plates) was erected, together with two batch fractionators suitable for the distillation of 10-gallon charges and equipped with columns equivalent to forty theoretical plates. All these columns were carefully tested to determine the number of theoretical plates, maximum boil-up rate, heat loss, etc. A stock of suitable naphtha cut was provided, and a series of experiments carried out on the continuous column with the double object of preparing sufficiently large samples of isoheptane for detailed test and examining the effect of various operating conditions. Tests, which are described later, were devised for analysing the products in the overlap region—*e.g.*, when operating the column at a cut point in the region of 70° C., to determine the amount of *n*-hexane in the bottoms and the amount of methylcyclopentane and benzene lost in the overhead.

The general shape which the process would assume in the refinery now began to emerge. It was evident that the crude distillation units would have to be operated to produce two base cuts, referred to as isohexane base and isoheptane base respectively; and it was certain that complete recovery of the two materials into the bases would not be achievable. The probable losses of isohexane and isoheptane due to poor cutting on the crude benches (which were not designed for such an operation as this) could be roughly estimated, but it was realized that the refinery would have to undertake an investigation on the full-scale units to establish optimum operating conditions. Each base so produced would have to be submitted to two stages of continuous fractionation, designated "Isohexane Op. 1" (removal of *n*-pentane overhead); "Isohexane Op. 2" (isohexane overhead; *n*-hexane and heavier bottoms); "Isoheptane Op. 1" (*n*-hexane and lighter overhead) and "Isoheptane Op. 2" (isoheptane overhead; *n*-heptane and heavier bottoms). Should an excessive loss of isoheptane have occurred due to its inclusion in the isohexane base, it could be recovered by passing the isohexane Op. 2 bottoms into isoheptane feed. The necessity for doing this would depend on the character of the bases and the desirability of achieving high overall recovery from the crude. Conservation of isohexane could be achieved by a reversal of this procedure.

DETAILED EXAMINATION OF THE COMPONENT PARTS OF ISOHEPTANE AND ISOHEXANE.

As soon as reasonably large samples of isohexane and isoheptane were available for test and detailed examination it became necessary to assess the way in which the various components of which they were made up affected the octane number, and particularly the rich mixture performance.

(a) *Isohexane.*

The constitution of isohexane was determined by fractionating it on the 2-litre scale using a 40-plate column. The true-boiling-point curve of a typical product is shown in Fig. 3, and the normal inspection data in Table II.

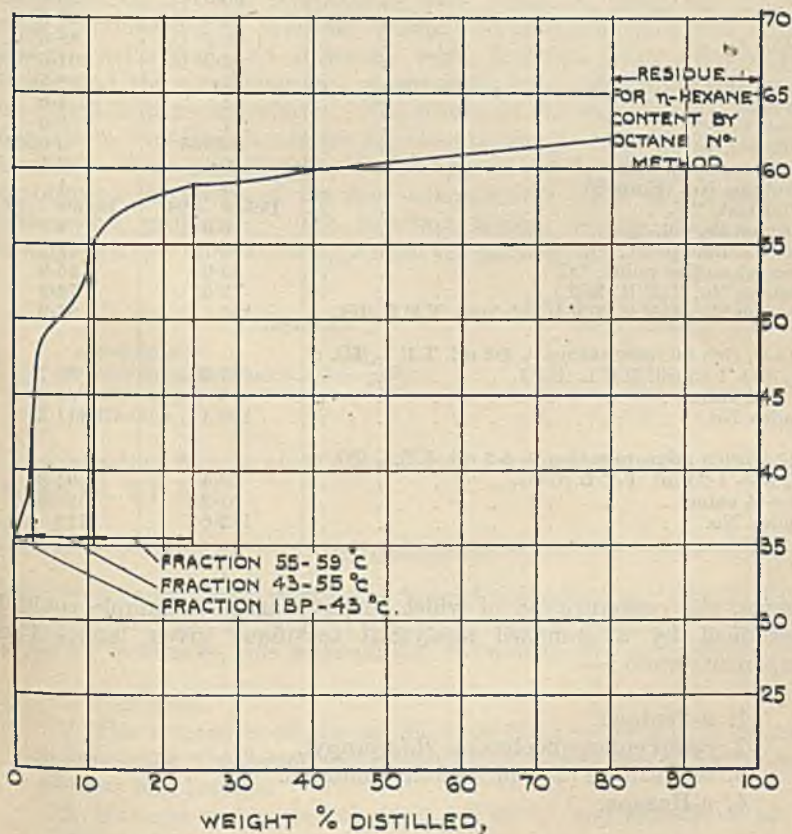


FIG. 3.

TRUE-BOILING-POINT DISTILLATION ROUTINE ANALYSIS—ISOHEXANE.

From a knowledge of the true-boiling-point curve and the employment of the conventional hydrocarbon-type analysis of the various cuts, it was possible to express the constitution of isohexane in terms of four com-

TABLE II.

Properties of Isohexane and Isoheptane.

	Isohexano.	Isoheptano.
Specific gravity at 60° F.	0.6670	0.7290
A.S.T.M. distillation :		
I.B.P.	57.5° C.	81.5° C.
5% distilling to	58.0° C.	82.5° C.
10% " " " " " " "	58.0° C.	83.0° C.
20% " " " " " " "	58.5° C.	83.5° C.
30% " " " " " " "	58.5° C.	84.0° C.
40% " " " " " " "	59.0° C.	84.5° C.
50% " " " " " " "	59.0° C.	85.0° C.
60% " " " " " " "	59.5° C.	85.5° C.
70% " " " " " " "	60.0° C.	86.0° C.
80% " " " " " " "	60.0° C.	87.0° C.
90% " " " " " " "	60.5° C.	88.5° C.
F.B.P.	62.0° C.	101.0° C.
Total distillate, vol.-%	98	98.5
Residue, vol.-%	1	1.0
Loss, vol.-%	1	0.5
Sulphur, wt.-%	0.0003	0.0002
Reid vapour pressure, lb./sq. in.	7.4	3.1
Bromine No. (Francis)	1	1
Cold test, ° F.	Below -94	Below -94
Aromatics, vol.-%	0.3	4.7
First aniline point, ° C.	68.7	51.1
Second aniline point, ° C.	69.0	55.8
Octane No. (C.F.R. MM.)	79.5	74.3
Octane No. (C.F.R. MM.) + 5.5 ml. T.E.L./IG.	103	91.9
F4 (3-C) rich mixture rating + 4.8 ml. T.E.L./IG. :		
% S + 1.25 ml. T.E.L./USG.	87.6	88.7
S + 4 value	86	86.8
Index No.	109.4	111.5
F4 (3-C) rich mixture rating + 5.5 ml. T.E.L./IG. :		
% S + 1.25 ml. T.E.L./USG.	89.4	91.2
S + 4 value	87.2	88.3
Index No.	112.6	116

ponents, the concentration of which in any particular sample could be determined by a standard analytical technique given later. These components were :—

1. *n*-Pentane.
2. *cyclo*Pentane/*neo*hexane/*diisopropyl*.
3. *iso*Hexanes (2- and 3-methylpentane).
4. *n*-Hexane.

Examination of a large number of samples of isohexane has demonstrated that the two middle components will not vary in properties or composition.

A more detailed examination, using infra-red analysis, allowed the composition to be expressed in terms of individual hydrocarbons as shown below :—

Constitution of Isohexane.

Hydrocarbon.	Wt.-%.	Vol.-%.	Wt.-% on Iranian crude.	Vol.-% on Iranian crude.
<i>n</i> -Pentane	3.0	3.2	0.04	0.05
<i>cyclo</i> Pentane	8.4	7.5	0.11	0.12
Neohexane	4.2	4.3	0.05	0.07
Diisopropyl	13.1	13.1	0.17	0.21
2- and 3-Methylpentanes	71.0	71.6	0.90	1.14
<i>n</i> -Hexane	0.3	0.3	0.004	0.005
	100.0	100.0	1.27	1.60

Samples of the four components were prepared, using the 10-gallon batch fractionators, in sufficient quantity to determine their rich mixture ratings. *n*-Pentane and *n*-hexane were prepared from suitable feed-stocks, and the remaining two components from isohexane produced on the 4-inch continuous column. The results of the engine tests were used to establish F4(3-C) rich-mixture blending factors (S + 4 scale), from which the rich mixture performance of a sample of isohexane of known weight composition (in terms of the four components) could be calculated by arithmetical proportion. The blending factors are as follows, and a detailed example of their use is given in the next section.

Component.	F4 (3-C) blending factor (S + 4 scale).
<i>n</i> -Pentane	40
<i>cyclo</i> Pentane/ <i>neo</i> hexane/ <i>diisopropyl</i>	127
<i>iso</i> Hexanes	75
<i>n</i> -Hexane	19

The blending factors given above were determined on the components plus 4.8 ml. T.E.L./IG.

 (b) *Isoheptane.*

The true-boiling-point curve of a typical isoheptane is shown in Fig. 4, and inspection data in Table II. Using a similar technique to that employed for isohexane, this material was reduced to five components:—

1. *n*-Hexane.
2. The remainder of the material distilling to 75° C. on the true-boiling-point distillation and consisting of a mixture of methyl*cyclo*pentane and benzene.
3. Material boiling in the range 75–85° C. and containing all the *cyclo*hexane.
4. Hydrocarbons distilling between 85° and 95° C.
5. Inferior diluent boiling above 95° C.

On this classification a typical analysis of isoheptane having a rich mixture performance of 88 (S + 4 scale) with 5.5 ml. T.E.L./IG. is as given below.

Constitution of Isoheptane.

Hydrocarbon boiling range.	Wt.-%.	Vol.-%.	Wt.-% on Iranian crude.	Vol.-% on Iranian crude.
<i>n</i> -Hexane	2.1	2.2	0.04	0.05
Aromatic and naphthenes boiling to 75° C.	12.1	11.3	0.23	0.25
Distilling in range 75-85° C.	25.4	24.7	0.48	0.54
Distilling in range 85-95° C.	59.5	60.9	1.13	1.34
Distilling above 95° C.	0.9	0.9	0.02	0.02
	100.0	100.0	1.90	2.20

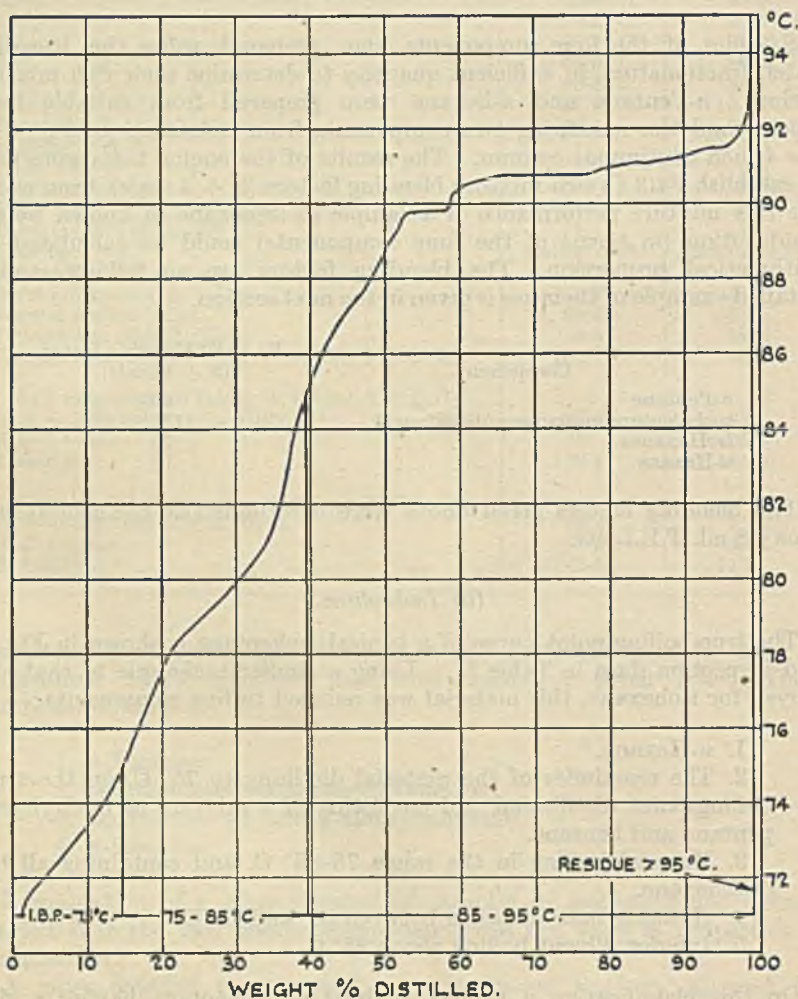


FIG. 4.

TRUE-BOILING-POINT DISTILLATION ROUTINE ANALYSIS—ISOHEPTANE.

Representative samples of the components were again produced on the 10-gallon batch-still and submitted to engine test to determine the F4(3-C) blending factors with 4.8 ml. T.E.L./IG.

Component.	3-C blending factor (S + 4 scale).
n-Hexane	20
Benzene and naphthene distilling to 75° C.	118
Distilling between 75 and 85° C.	108
Distilling between 85 and 95° C.	74
Distilling above 95° C.	48

The determination of each of these components is carried out in the manner described later in the section headed "Procedure for the Routine Analysis of Isoheptane."

(c) *Detailed Analysis of Isohexane.*

The procedure in analysing completely a sample of isohexane is as follows, where the figures given represent those actually obtained on a sample of isohexane of Iranian origin.

Two litres of isohexane were fractionated in a 40-plate helix packed column, and the following fractions were taken on the true-boiling-point distillation.

Fraction.	Boiling range of fraction, ° C.	Wt.-% of fraction.
1	I.B.P.-55	15.82
2	55 -59	10.14
3	59 -62.5	67.88
4	62.5-F.B.P.	6.16
		100.00

Fractions 1 and 2 were then refractionated through a 25-plate batch still and cuts taken on each at 43, 50 and 58.0° C. with the following results.

Fraction 1.

Boiling range, ° C.	Wt.-% of cut.	Wt.-% on original charge.	Density 20° C.	R.I. 20° C.
I.B.P.-43	19.01	3.01	0.6278	1.3585
43-50	57.61	9.11	0.7134	1.3941
50-58	6.79	1.07	0.6882	1.3849
58-F.B.P.	16.59	2.63	0.6664	1.3732
	100.00	15.82	—	—

Fraction 2.

Boiling range, ° C.	Wt.-% of cut.	Wt.-% on original charge.	Density 20° C.	R.I. 20° C.
48.5-50.0	14.88	1.51	0.7074	1.3921
50.0-58.0	16.37	1.66	0.6807	1.3821
58.0-60.5	62.76	6.36	0.6575	1.3727
60.5-F.B.P.	5.99	0.61	—	1.3740
	100.00	10.14	—	—

The fractions obtained from these distillations excluding fraction 4, the residue, were then bulked to give five fractions for infra-red analysis, and the results of this analysis are given below.

Boiling Range, ° C.	I.B.P.-43.0	43.0-50.0	50-58.0	58.0-60.5+	59.0-62.5
Wt.-% on original	3.01	10.62	2.73	9.60	67.88
Composition, wt.-% :					
<i>n</i> -Pentane	99	—	—	—	—
<i>cyclo</i> Pentane	0.1	68.1	46.3	—	—
<i>neo</i> Hexane	—	31.9	28.1	Trace	—
Diisopropyl	—	—	11.5	27.3	15.1
2-Methylpentane	—	—	14.1	72.7	64.7
3-Methylpentane	—	—	—	Trace	20.2

Summation of these gives the following composition for the original isohexane.

Hydrocarbon.	Wt.-%.
<i>n</i> -Pentane	3.0
<i>cyclo</i> Pentane	8.4
<i>neo</i> Hexane	4.2
Diisopropyl	13.1
2-Methylpentane	51.4
3-Methylpentane	13.7
Residue (Fraction 4)	6.2
	100.0

The residue, Fraction 4, was charged to a 25-plate spiral screen batch still and distilled at a reflux ratio of 40 to 1. The percentage of *n*-hexane present was calculated from the distillation curve by assuming the concentration of *n*-hexane to increase linearly from 0 to 100 per cent. over the boiling range 3-methylpentane (b.pt. 63.3° C.) and *n*-hexane (b.pt. 68.8° C.), and by this method a figure of 0.3 per cent. weight for the *n*-hexane content of the original charge was obtained. From the shape of the true-boiling-point curve of the residue the remainder, 6.2-0.3—*i.e.*, 5.9 per cent.—was assumed to be a mixture of 2- and 3-methylpentanes.

This allows the final composition of the isohexane to be expressed as follows.

Hydrocarbon.	Wt.-%.	Vol.-%.
<i>n</i> -Pentane	3.0	3.2
<i>cyclo</i> Pentane	8.4	7.5
<i>neo</i> Hexane	4.2	4.3
Diisopropyl	13.1	13.1
2- and 3-Methylpentanes	71.0	71.6
<i>n</i> -Hexane	0.3	0.3
	100.0	100.0

(d) Procedure for Routine Analysis of Isohexane.

The results of detailed analyses on isohexane have shown the existence of a constant ratio between *cyclopentane*, *neohexane*, and *diisopropyl*. Referring to the table above, it is seen that the amounts of these three

components are in the ratio 8.4 to 4.2 to 13.1 (weight per cent. figures) which expressed as a percentage becomes 33 to 16 to 51. These ratios are important in the interpretation of the subsequent analysis.

A weighed quantity, about 2 litres, of isohexane is distilled in a 40-plate batch still to an overhead temperature of 43.0° C. using a reflux ratio of 40 to 1. The distillation is then stopped and the loss in weight reckoned as the *n*-pentane content of the sample.

The distillation is continued at 20 to 1 reflux ratio until about 80 per cent. vol. has been taken overhead, taking fractions at temperatures of 55° C. and 59° C. The density and refractive index at 20° C. are then determined on the two fractions I.B.P.-55° C. and 55-59° C., and specific refractions are calculated.

Using specific refractions of 0.35034 for *n*-pentane and 0.33549 for a blend of 67 per cent. *cyclopentane*—33 per cent. *neohexane*, the composition of the first fraction, I.B.P.-55° C.—is calculated by linear interpolation.

The composition of the fraction 55-59° C. is now calculated in terms of a mixture of 67/33 *cyclopentane-neohexane* and *diisopropyl 2- and 3-methylpentanes*, using a value of 0.34735 for the specific refraction of this latter mixture.

The total amount of the *cyclopentane-neohexane-diisopropyl* fraction in the sample is then taken as the *cyclopentane-neohexane* content multiplied by 100/49.

The octane number of the residue, approximately 20 per cent. volume on the charge, is then compared directly with the octane number of a specially prepared *n*-hexane-free residue of similar boiling range. This allows the *n*-hexane content of the residue to be calculated on the basis that 1 per cent. weight of *n*-hexane is equivalent to 0.4 difference in octane number. This method of estimating the *n*-hexane content of the sample is quicker than the one described in the preceding section, and gives the same results.

The fourth component, the 2- and 3-methylpentanes, is obtained by difference. An example showing the method of calculation is given below, the corresponding true boiling point curve being Fig. 3.

The rich mixture performance as calculated, 85, agrees well with the determined value (see Table II) of 86.

Calculation of F4(3-C) Performance for Isohexane.

Distillation analysis (true boiling point) :—	
Fraction I.B.P.-43° C. :	
% wt. of sample	2.44
Fraction I.B.P.-55° C. :	
% wt. of sample	10.22
Density at 20° C.	0.6856
Refractive index at 20° C.	1.3830
Specific refraction	0.34023
Fraction 55-59° C. :	
% wt. of sample	14.06
Density at 20° C.	0.6693
Refractive index at 20° C.	1.3782
Specific refraction	0.34462
Residue :	
% wt. of sample	17.56
Octane No. (C.F.R. M.M.)	75.1
Octane No. (C.F.R. M.M.), <i>n</i> -hexane free standard	75.8

Calculation of F4(3-C) Performance for Isohexane.—Continued.

Composition and 3-C Performance :—

	Wt.-%.	F4(3-C) factor.	Calc. F4(3-C) perf.
n-Pentane	2.44	40	1.0
cycloPentane/neoHexano/diisopropyl	20.82	127	26.4
isoHexanes	76.43	75	57.3
n-Hexane	0.31	19	0.1
	100.00	—	84.8

(e) Procedure for Routine Analysis of isoheptane.

A 2-litre charge of isoheptane is distilled in a 40-plate batch still using a reflux ratio of 20 : 1 and the weight percentages distilling to 75° C. and 85° C. are recorded. The fraction to 75° C. is analysed for its aromatic content by acid adsorption, and the naphthene and paraffin content of the acid-washed product is determined by the specific refraction method. In making this calculation it is assumed that the aromatic-free material is a binary mixture of normal hexane with a specific refraction of 0.34724 and methylcyclopentane, specific refraction 0.33081. The residue from the 40-plate column distillation, representing about 10 per cent. on the charge, is redistilled in a small 25-plate column and the percentage distilling above 95° C. is recorded. An example of such an analysis follows, while the true-boiling-point curve applying to this particular case is given in Fig. 4.

Calculation of F4(3-C) Performance for Isoheptane.

Distillation Analysis (true boiling point) :—

Fraction I.B.P.—75° C. :

% wt. of sample	14.21
Density at 20° C.	0.7615
Refractive index at 20° C.	1.4224
Aniline point, ° F.	65.3
Absorption in sulphuric acid, vol.-%	23.2
wt.-%	26.2

Fraction " I.B.P. " — 75° C. (aromatic free) :

Density at 20° C.	0.7326
Refractive index at 20° C.	1.4042
Aniline point, ° F.	105.4
Specific refraction	0.33397

Composition of original fraction :

Aromatic content, wt.-% of fraction	24.1
Naphthene content, wt.-% of fraction	61.3
Paraffin content, wt.-% of fraction	14.6

Fraction 75–85° C. :

% wt. of sample	25.42
-----------------	-------

Fraction 85–95° C. :

% wt. of sample	59.51
-----------------	-------

Fraction above 95° C. :

% wt. of sample	0.86
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Composition and 3-C Performance :—

	Wt.-%.	F4(3-C) factor.	Calc. F4(3-C) perf.
n-Hexane	2.10	20	0.4
Benzene and naphthene distilling to 75° C.	12.11	118	14.3
Distilling between 75–85° C.	25.42	108	27.5
Distilling between 85–95° C.	59.51	74	44.0
Distilling above 95° C.	0.86	43	0.4
	100.00	—	86.6

PRELIMINARY INVESTIGATION OF PLANT CONTROL.

The experiments on the semi-scale continuous column allowed methods of controlling the operation of the plant to be investigated in some detail. Although these methods were for the most part decided upon in detail at this stage, they will not be discussed further here, since they are more suitably included in the consideration of the full-scale plant. It was also possible to obtain further information from the operation of the 50-plate column in the refinery, which produced isoheptane at an average daily rate of 30,000 g.p.d. for about a year, before the full-scale plant was commissioned.

MANUFACTURING SPECIFICATIONS FOR UNREFINED PRODUCTS.

The setting up of manufacturing specifications necessitated striking a compromise between the plant requirements for the production of high-quality material, the overall recovery desired, and the amounts of unwanted degrading components which could be tolerated. All the data were now available for striking this balance, and as a result it was decided to manufacture to the following specifications after the addition of 4.8 ml. T.E.L/IG.

Isohexane—calculated F4(3-C) rich mixture rating, following procedure discussed above for routine analysis: 84.

Isoheptane—calculated F4(3-C) rich mixture rating, following procedure discussed above for routine analysis: 87.

In the case of isoheptane the amount boiling above 95° C. was restricted to 5.0 per cent. maximum, in order to ensure that the refinery production lay inside the range of compositions on which the extensive preliminary work had been carried out.

ESTIMATES OF PLANT REQUIREMENTS.

Concurrently with the latter stages of the work so far described, consideration was given to the plant which would be required to manufacture the products on the full scale. The results of the various experiments on the forty-three theoretical plate 4-inch column, combined with the experience which had been gained in the refinery in operations on the 50-plate column and the characteristics of the base-stocks which would be produced on the crude units, were used as the starting point for calculating the number of plates and reflux ratios to be used for the various operations. The calculations were not very precise, and it was necessary to allow a considerable factor of safety, particularly in regard to the number of actual plates in the various columns.

The scale on which the operation was to be carried out was also fixed. The ultimate target was to manufacture at the rate of 1,000,000 tons per year of finished 100 octane number aviation spirit, this figure having been arrived at from the expected throughput of the refinery and the estimated yields of the various components which would be obtained. To combine balanced production and erection of plant for this and other processes, this figure was to be approached in two stages.

The first stage comprised the following plant as far as the superfractionation equipment was concerned; all the columns being 12 feet in diameter.

Isohexane, Op. 1	1- 76-plate column.
Isohexane, Op. 2	1-100-plate column.
Isoheptane, Op. 1	1-100-plate column.
Isoheptane, Op. 2	1- 76-plate column.

Depending on the combination of crude benches employed and on whether maximum isohexane or maximum isoheptane was in demand, the expected yields from this combination of units varied between 93,000 and 111,000 g.p.d. of unrefined isohexane, together with 119,000 and 158,000 g.p.d. of unrefined isoheptane.

It was realized that the allocation of a 76-plate column for isohexane, Op. 1, was generous, and when two further 100-plate units were installed, this fact, combined with the additional knowledge that both isohexane, Op. 2, and isoheptane, Op. 2, could be carried out with fewer plates, led to a reallocation of the superfractionators. In this reallocation the additional two 100-plate units were converted into 50-plate units, as will be explained later, giving a final disposition as follows:—

Isohexane, Op. 1	1- 50-plate column.
Isohexane, Op. 2	2- 76-plate column.
Isoheptane, Op. 1	2-100-plate column.
Isoheptane, Op. 2	2- 50-plate column.
Spare unit	1- 50-plate column.

The function of the spare unit was either to act as a standby or as an intermediate column operating on isohexane, Op. 2 bottoms, so as to remove normal hexane overhead and enable the residue containing isoheptane constituents to be passed to isoheptane base. Operating in this manner the expected yields of isohexane and isoheptane were 154,000 and 228,000 g.p.d., respectively.

The detailed description of the full-scale units, which comprises the second half of this paper, has been based on the operation of the first stage for two reasons. First, each operation is confined in this case to one column, and the complication introduced by operating two columns in parallel on some of the operations, which is not important from the purely distillation point of view, is avoided. Secondly, the main commissioning troubles were overcome at this stage and, as would be expected, more knowledge of the performance of the plant was acquired in the course of overcoming them than was obtained when extending the operation to the larger number of units.

PART II.—PRODUCTION OF ISOHEXANE AND ISOHEPTANE IN ABADAN.

This section deals with the plant required and the procedure to be followed when producing superfractionated products on the scale mentioned previously. It is appreciated that any plant layout is dependent on local conditions, but it is thought that a brief description of the refinery flow-sheet incorporating superfractionation, which has actually operated on a production basis, will help in grasping the main points involved, and Fig. 5 illustrates the scheme as operative in Abadan during August 1943.

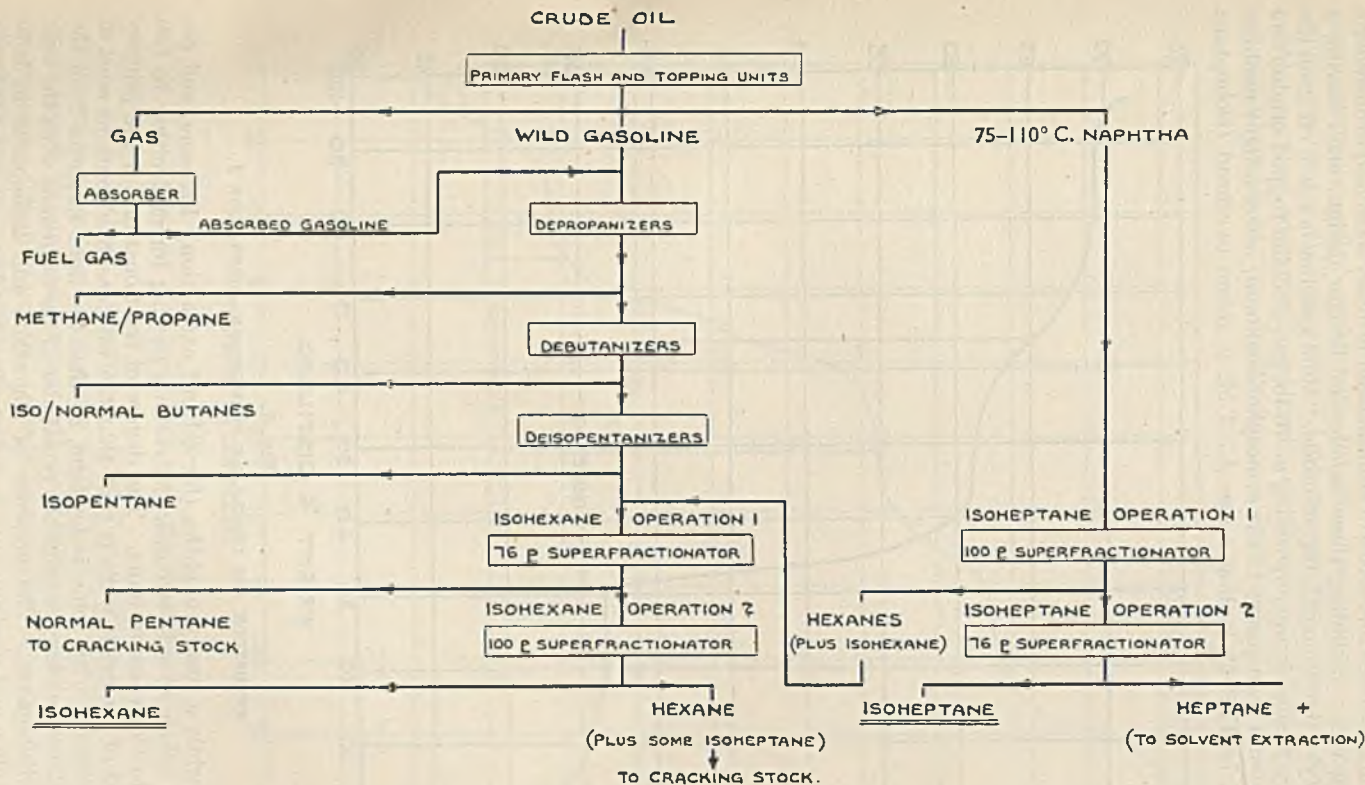


FIG. 5.

REFINERY FLOWSHEET, INCORPORATING SUPERFRACTIONATION.

(The above scheme is representative of operation in Abadan, August 1943.)

Crude oil passes to a series of combination primary flash and atmospheric topping units, including three benches of Badger design, each having a capacity of 3 million gal./day of crude. Wild gasoline is taken off from the primary flash columns, operating at 70-lb. ga., and the topped crude from this column then passes to the atmospheric column, where a light naphtha cut of 75–110° C. boiling range A.S.T.M. is taken overhead (isohexane

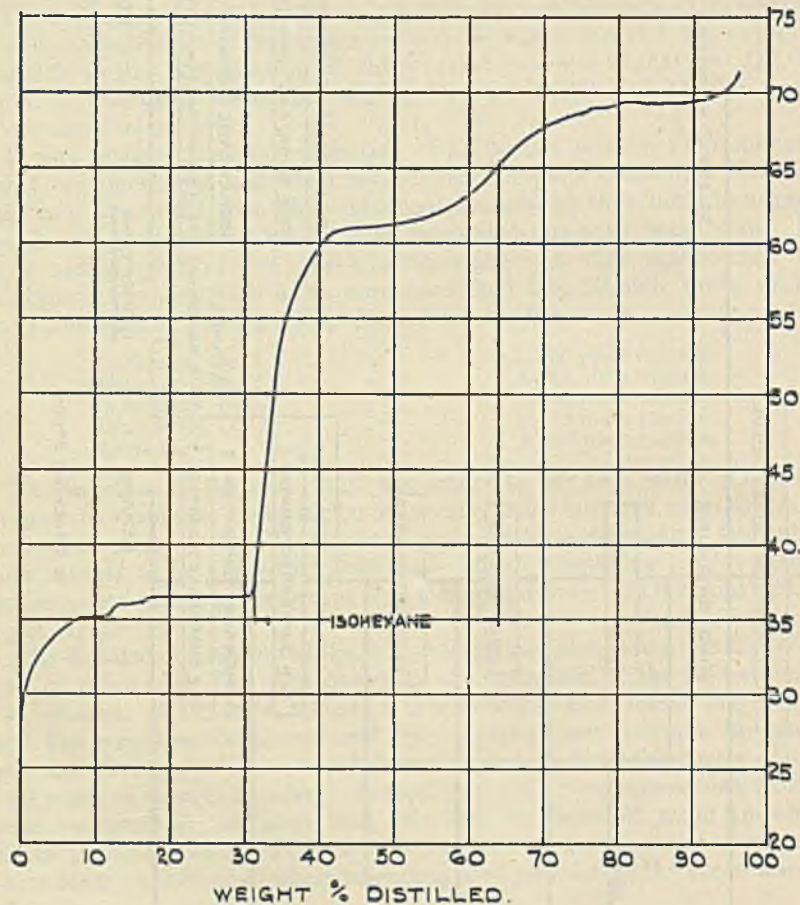


FIG. 6.

FEEDSTOCK FOR ISOHEXANE PRODUCTION—OPERATION 1.

base), while a heavy naphtha (for solvent extraction), kerosine, and gas oil are taken as side streams, the residue passing forward either to fuel oil or to the vacuum units. The degree of fractionation on the primary columns of the crude units is such that the wild gasoline contains most of the isohexane with some isoheptane, so that the light naphtha from the atmospheric column contains the remainder of the isohexane with the bulk of the isoheptane. The presence of isohexane and isoheptane in both the gasoline and naphtha cuts is unfortunate, but was unavoidable in the

scheme under discussion, on account of the impossibility of producing and processing such a wide single cut from the primary columns which would enable the full recovery of both components. This is a function of the installed equipment, and could only be remedied by the provision of crude benches having a higher degree of fractionation.

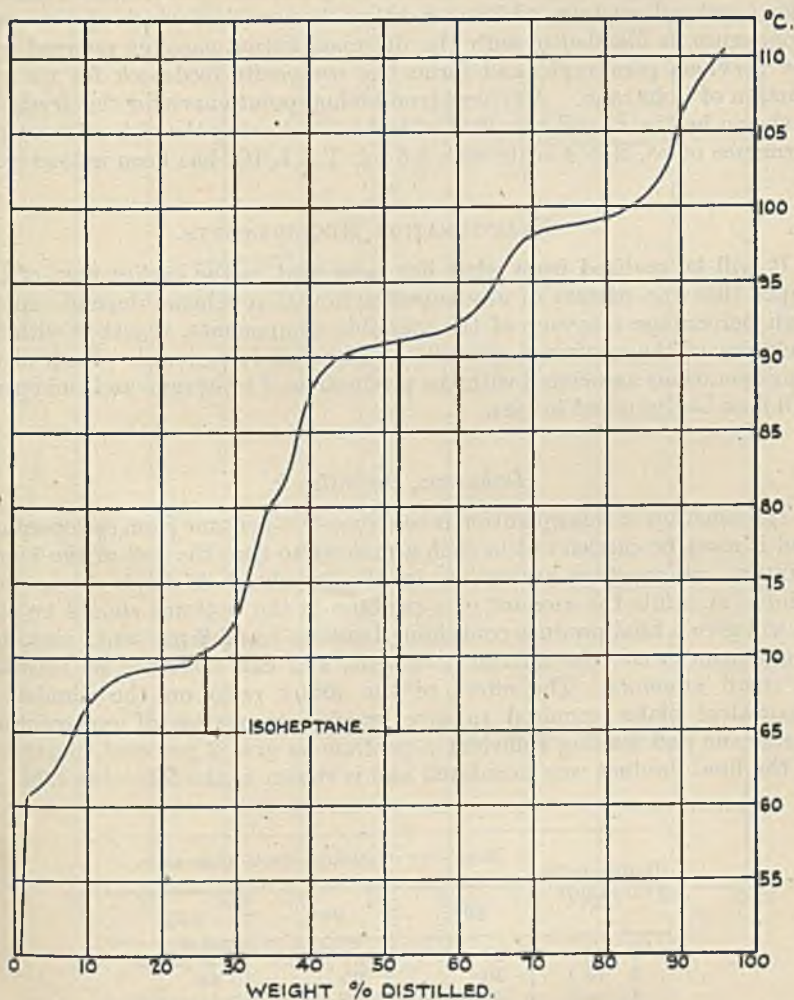


FIG. 7.

FEEDSTOCK FOR ISOHEPTANE PRODUCTION—OPERATION 1.

The wild gasoline, representing about 7 per cent. vol. on crude, then passes to the depropanizers, where a C_1/C_3 fraction is removed, and this is followed by debutanization and deisopentanization in conventional equipment. The bottoms from the deisopentanizer form part of the feedstock for isohexane production.

The isoheptane base from the atmospheric column, equivalent to 6-7 per cent. vol. on crude, is the feedstock used in the preparation of isoheptane, and a typical true-boiling-point curve is illustrated in Fig. 7, which also shows the yield of isoheptane, having the rich mixture performance of 89, S + 4 scale, with 5.5 ml. T.E.L./IG. The isoheptane base is then processed through a superfractionator for the removal of hexane overhead. This overhead product, which is mostly *n*-hexane, but which also contains isohexanes, is blended in with the deisopentanized gasoline referred to in the previous paragraph, and forms the composite feedstock for the production of isohexane. A typical true-boiling-point curve for this feedstock is shown in Fig. 6, and the yield of isohexane having the rich mixture performance of 88, S + 4 scale with 5.5 ml. T.E.L./IG. has been indicated.

FRACTIONATION REQUIREMENTS.

It will be realized from what has been said in the earlier part of this paper that the success of any superfractionation scheme depends on the high percentage recovery of the valuable components, together with the inclusion of the minimum quantities of low-grade material. Each of the four operations associated with the production of isohexane and isoheptane will now be discussed in turn.

Isohexane, Operation 1.

The function of this operation is to separate *n*-pentane from *cyclopentane*, and it must be carried out in such a manner so that the loss of the binary mixture *cyclopentane-neohexane* in the overhead is kept down to a minimum, while the amount of *n*-pentane in the bottoms should be such as will give a final product containing between 1 and 2 per cent., since this component is not too inferior a diluent, and can therefore be tolerated in small amounts. The effect of the reflux ratio on the number of theoretical plates required to give varying recoveries of *cyclopentane-neohexane* and leaving sufficient *n*-pentane to give 2 per cent. maximum in the final product was calculated and is shown in the following table:—

Reflux ratio (internal).	Recovery of <i>cyclopentane-neohexane</i> .		
	80%.	90%.	95%.
6 to 1	27 plates	31 plates	35 plates
5 to 1	30 "	35 "	40 "
4 to 1	37 "	45 "	51 "
3.5 to 1	47 "	58 "	—

This operation has been carried out in Abadan with a feedstock substantially as indicated in Fig. 6, using a 76-plate column and operating with a cold reflux ratio of $3\frac{1}{2}$ to 1, which results in the recovery of 90-95 per cent. of the *cyclopentane-neohexane* fraction into the bottoms product with a *n*-pentane content of 0.5 per cent.

Isohexane, Operation 2.

The bottoms from isohexane, Operation 1 forms the feedstock for operation 2, and the true-boiling-point curve for this material is shown in Fig. 8. The success of this operation is dependent on the production of an overhead product substantially free from *n*-hexane. Referring to Fig. 8 it is seen that a cut point on the true-boiling-point curve for this operation has been

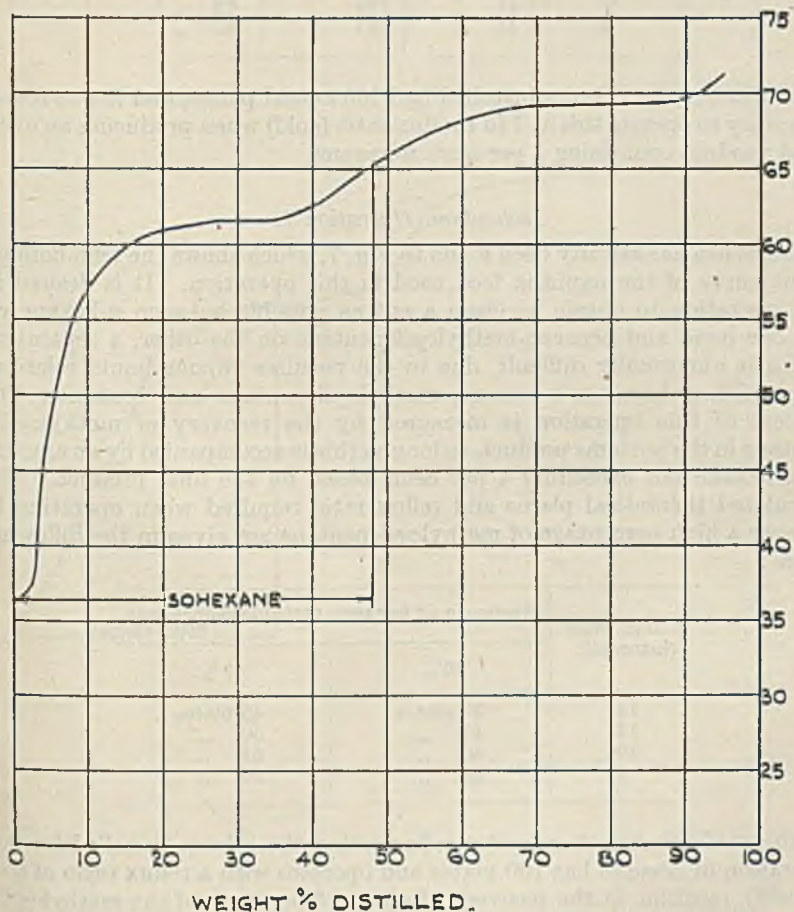


FIG. 8.

FEEDSTOCK FOR ISOHEXANE PRODUCTION—OPERATION 2.

located at 65° C. Lowering this cut point would result in the production of a higher-quality product with sacrifice of yield, while raising it would speedily result in the inclusion of excessive quantities of *n*-hexane, with a consequent rapid drop in quality, out of all proportion to the slight increase in yield obtained.

With the cut point on the true-boiling-point curve as indicated in Fig. 8, the theoretical plates and reflux ratio calculated to produce an overhead product of low *n*-hexane content are shown in the table given below:—

Reflux ratio (internal).	% <i>n</i> -hexane in overhead product.		
	3.0.	1.5.	0.5.
12	34 plates	48 plates	58 plates
11	36 "	50 "	62 "
10	38 "	53 "	66 "
9	41 "	58 "	73 "
8	45 "	67 "	87 "

In Abadan, the column installed had 100 actual plates, and it was found necessary to operate this at 7 to 1 reflux ratio (cold) when producing an overhead product containing 1 per cent. *n*-hexane.

Isoheptane, Operation 1.

Reference has already been made to Fig. 7, which shows the true-boiling-point curve of the naphtha feed used in this operation. It is desired in this operation to obtain as clean a cut as possible between *n*-hexane on the one hand and benzene-methylcyclopentane on the other, a separation which is abnormally difficult, due to the peculiar vapour liquid relationship existing between *n*-hexane, methylcyclopentane and benzene. The success of this operation is measured by the recovery of methylcyclopentane in the bottoms product, so long as this is accompanied by an amount of *n*-hexane not exceeding 4 per cent. based on the final product. The calculated theoretical plates and reflux ratio required when operating to recover a high percentage of methylcyclopentane are given in the following table:—

Reflux ratio (internal).	Recovery of benzene-methylcyclopentane.	
	80%.	85%.
14	39 plates	45 plates
12	41 "	50 "
10	46 "	61 "
9	52 "	80 "

To avoid running at excessive reflux ratios, the column installed for this operation in Abadan has 100 plates and operates with a reflux ratio of 6 to 1 (cold), resulting in the recovery of about 85 per cent. of the methylcyclopentane into the bottoms product, together with 1 per cent. of *n*-hexane.

Isoheptane, Operation 2.

The true-boiling-point curve of the feedstock is reproduced in Fig. 9, and the operation of this operation is the production of the overhead product containing a minimum of material boiling above 95° C. (*n*-heptane). In Fig. 9 a cut point on the true-boiling-point curve has been located at 92° C. Lowering this cut point would, as was the case with isohexane, result in the production of a higher-quality product with sacrifice of yield. On the other hand, it will be observed that an increased yield of a slightly

lower quality product could be achieved before approaching the boiling point of pure *n*-heptane at 98° C. In other words, the chosen cut point for specification *isoheptane* lies on a flat in the 92–93° C. boiling range, a fact which makes this separation considerably easier than *isohexane*, Operation 2.

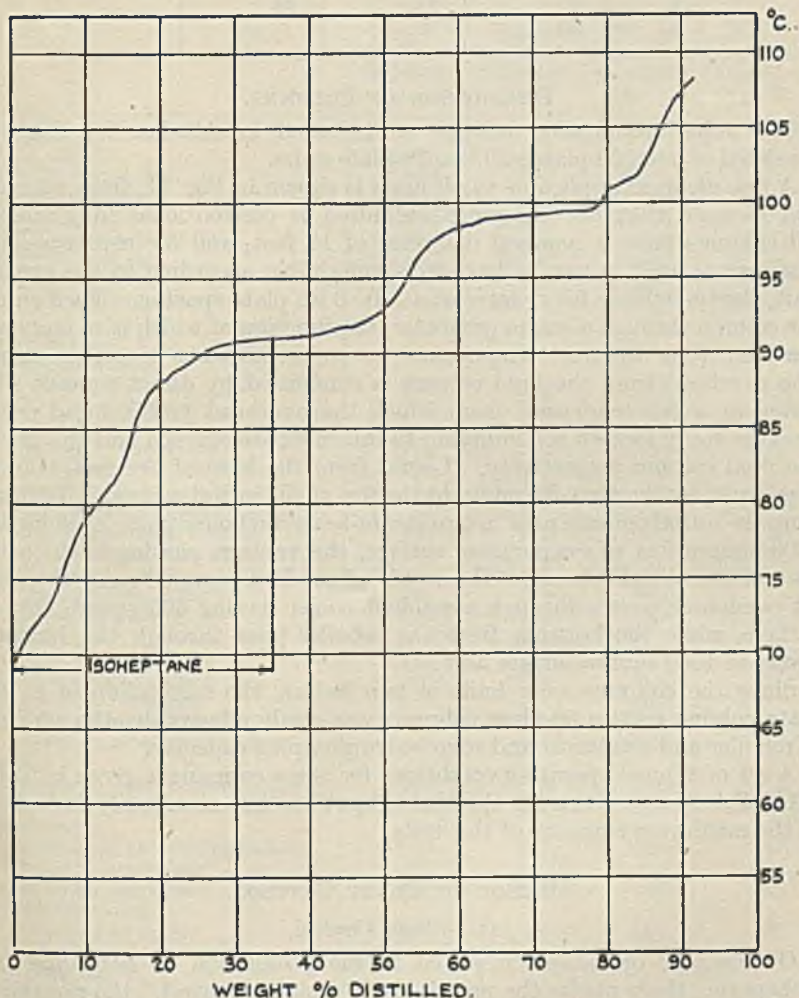


FIG. 9.

FEEDSTOCK FOR ISOHEPTANE PRODUCTION—OPERATION 2.

In Abadan a 76-plate column is used for this separation with a reflux ratio (cold) of 3.5 to 1, giving an overhead product containing 1–2 per cent. of material boiling above 95° C. The theoretical relationship between the number of plates required for the separation, the reflux ratio, and percentage boiling above 95° C. is given below :—

Reflux ratio (internal).	% Boiling in overhead above 95° C.			
	5.0.	3.0.	1.5.	0.5.
7	17 plates	24 plates	34 plates	45 plates
6	19 "	27 "	40 "	54 "
5	21 "	33 "	56 "	—
4	43 "	—	—	—

DESCRIPTION OF COLUMNS.

The superfractionation scheme as installed in Abadan for Stage I consisted of two 100-plate and two 76-plate units.

A line diagram applicable to all units is shown in Fig. 11, from which it will be seen that the equipment required is conventional in principle. All columns have a nominal diameter of 12 feet, and for constructional reasons are built in two halves, each containing, according to the type of unit, thirty-eight or fifty plates at a 2 ft. 6 in. plate spacing. Feed enters the column through a steam preheater, the function of which is to maintain the feed at a constant temperature so as to minimize metering errors. The overhead from the final column is condensed by direct contact with water in a jet condenser, from which the overhead product and reflux streams are drawn off for pumping to intermediate storage and the top of the final column respectively. Liquid from the base of the final column is pumped by the transfer pump to the top of the initial column. Bottoms from the initial column pass to two steam-heated reboiler units, each having 3000 square feet of evaporating surface, the vapours passing back to the base of the initial column. The overhead product pumped away from the jet condenser passes through a product cooler having 500 square feet of surface, while the bottoms from the reboiler pass through the bottoms cooler of 1060 square surface area.

Since the columns were built in two halves, the conversion of a 100-plate column to two 50-plate columns was easily effected by the addition of reboiler and condenser and some extra pumping capacity.

A set of typical operating conditions for these columns is given in Table III. The figures given for the throughput are not necessarily indicative of the maximum capacity of the units.

METHOD OF PLANT CONTROL.

(a) *Volume Control.*

Of the four operations involved in the production of isohexane and isoheptane, three utilize the principle of "volume control," the remaining one being controlled by the temperature recorded at a certain plate in the tower.

It is usual to control the operation in fractionating towers by varying the amount of reflux to the column, so as to maintain a constant temperature at the column head or at some intermediate point in the column. Precision in fractionation is not possible, however, by such a method of control when the boiling ranges of the products to be obtained are separated by small temperature differences.

TABLE III.

Actual Plant Operating Conditions—Production of Isohexane and Isoheptane.

	Isohexane production.		Isoheptane production.	
	No. 1 superfractionator, Op. 1.	No. 2 superfractionator, Op. 2.	No. 3 superfractionator, Op. 1.	No. 4 superfractionator, Op. 2.
Quantities :				
Feed, gal./hr.	20,800	10,900	21,000	22,800
Overhead, gal./hr.	7,960	4,950	6,850	6,650
Bottoms, gal./hr.	12,840	5,950	14,150	16,150
Reflux, gal./hr.	31,800	35,800	40,400	29,000
Total overhead, gal./hr.	39,760	40,750	47,250	35,650
Transfer, gal./hr.	59,400	38,200	72,000	47,800
Steam, lb./hr.	45,850	43,000	61,300	60,000
Water, gal./hr.	47,800	56,600	71,000	65,500
Temperatures (° F.) :				
Feed preheater :				
Feed to preheater	109	108	106	107
Feed ex preheater	130	138	134	132
Column :				
Vapour to condenser	152	140	150	182
Vapour above plate 53 or 69	(53) 160	(69) 165	(69) 179	(53) 212
Vapour above plate 47 or 61	(47) 162	(61) 164	(61) 183	(47) 218
Vapour above plate 39 or 51	(39) 174	(51) 171	(51) 197	(39) 226
Vapour ex initial column	180	175	201	230
Vapour above plate 25 or 34	(25) —	(34) 180	(34) 214	(25) 248
Vapour above plate 13 or 16	(13) 190	(16) 190	(16) 244	(13) 259
Vapour above plate 1	222	198	247	—
Liquid ex plate 1	223	198	248	—
Reboilers :				
Vapour ex reboiler 1	225	202	254	276
Vapour ex reboiler 2	225	200	254	276
Liquid to reboiler 1	—	—	248	—
Liquid to reboiler 2	—	—	248	—
Liquid ex reboilers 1 and 2	226	202	256	278
Reflux				
Reflux to final column	129	120	126	123
Transfer to initial column	178	171	199	226
Condenser :				
Condensate to overhead cooler	125	121	126.5	125
Water to condenser	71	68.5	77	76
Water ex condenser	126	118	130	121
Overhead cooler :				
Overhead ex cooler	100	105	105	108
Water ex cooler	—	106	106	—
Bottoms cooler :				
Bottoms to cooler	—	—	256	274
Bottoms ex cooler	76	71	206	79
Water ex cooler	—	73	119	—
Pressures (lb./sq. in. ga.) :				
Column top	28	2	2	1
Final column base	—	7.5	10.5	6.5
Initial column top	—	7.5	13	8.5
Initial column base	32.5	13.5	24	20

TABLE III—continued.

	Isohexano production.		Isoheptane production.	
	No. 1 superfrac- tionator, Op. 1.	No. 2 superfrac- tionator, Op. 2.	No. 3 superfrac- tionator, Op. 1.	No. 4 superfrac- tionator, Op. 2.
Steam :				
Main steam, lb./sq. in. ga.	216	211	214	218
Main steam, ° F.	—	—	442	445
Steam to reboilers, lb./sq. in. ga.	20	—	83	70
Condensate ex reboiler, lb./sq. in. ga.	—	—	35	—
Condensate ex reboiler 1, ° F.	—	—	276	—
Condensate ex reboiler 2, ° F.	—	—	264	—
Position of feed point, plate no.	39	51	51	15
Reflux to top tray, lb./hr.	199,800	240,000	273,000	211,800
Reflux from top tray, lb./hr.	217,000	255,000	298,000	255,500
Vapour velocity below top tray, ft./sec.	1.4	3.2	4.7	3.3
Heat input to reboilers, M.B.Th.U./hr.	45.8	46.4	60.1	55.6
Heat input to preheaters M.B.Th.U./hr.	1.5	1.7	2.2	2.0

The principle on which volume control is based is the control of the quantity of one of the product streams withdrawn from the column as a fixed proportion of the feed, the quantity of the product so withdrawn being determined by a true-boiling-point analysis of the feed. Either the bottoms or overhead streams may be controlled in this manner, the other product being removed from the column at such a rate as to prevent accumulation in the tower. Where the feed composition varies, as will be indicated by successive analyses of tanks of feedstock, small adjustments to the controls on the column are made with the process in operation.

So long as accurate metering and adequate tankage are insisted upon, the control of superfractionators using this principle has proved simple and efficient.

(b) Tankage.

Since of the four operations involved in manufacturing isohexane and isoheptane three employ the process of volume control, the greatest care must be given to the selection of suitable tankage when laying out a scheme for the preparation of these components. The layout as finally adopted must satisfy the following points. The tank capacity must be such as to allow a true-boiling-point distillation of the tank contents to be made, where this is necessary, prior to feeding to the unit. Tanks should be top filling and bottom emptying, so as to minimize the effects of layering.

A brief description of the tankage system as used in Abadan with satisfactory results over a period of three years will help in understanding the significance of these points.

The feedstock for isohexane, Operation I is drawn alternately from one of two 2,500,000-gal. tanks. Experience has shown that it is unnecessary

to analyse the contents of these tanks, once a steady scheme of operation has been reached, since the application of temperature control to this process will take care of minor fluctuations in feed composition. Nevertheless, it is thought desirable that two feed-tanks should be used, as this minimizes the effects of layering, and, moreover, provides flexibility if it should become necessary to make a substantial alteration in the composition of the base due to changes in process operations elsewhere in the refinery.

The bottoms from isohexane, Operation 1 pass into one of a further two 2,500,000-gal. tanks. These operate on a cycle, whereby, as one is filled and analysed, the other is being processed through the second column to give the unrefined isohexane. While it is possible that one of these might be dispensed with, it is believed that an appreciable loss in yield would result, due to the unavoidable introduction of a safety factor to cover possible variations in the quality of the feedstock and the consequent production of an off-grade product.

Feedstock for isoheptane, Operation 1 is taken from one of three 2,500,000-gal. tanks operating in sequence. As opposed to isohexane, Operation 1, it is essential to analyse each tank as filled in order to determine the percentage overhead to be taken from the column. The three tanks are so arranged that while one is receiving base, a second is feeding the unit, while the third is being analysed. For a unit processing 500,000 gal. day, a capacity of 2,500,000 gal. is very suitable, giving a time cycle of four days to fill, four days to process, and allowing four days to analyse the third tank.

Experience has shown that as long as the naphtha feed is fairly constant in quality it is unnecessary to analyse the bottoms from isoheptane, Operation 1 before proceeding with Operation 2, and the two superfractionators engaged on these respective duties in Abadan have run in series on occasion in a perfectly satisfactory manner. This practice was discontinued for two reasons: first, a temporary upset on the first unit was immediately passed on to the second, while secondly, a short shutdown on one unit would automatically cause a loss of product from the second. This method of operation cannot be recommended, and in actual practice a balance tank was employed but, in general, no intermediate analysis was carried out.

As regards the storage necessary for the unrefined, the acid washed, and re-run acid washed materials, it is recommended that this should be such as to permit the testing of tanks of finished material prior to the final blending into aviation gasoline, especially if the process is being run so as to give no margin on product quality. Moreover, adequate storage at this stage permits of occasional meter check-ups which are frequently of great use.

(c) *Instrumentation.*

The importance of accurate instrumentation in all superfractionation processes cannot be over-estimated. In order to prevent misunderstanding, it must be stated that an accurate and absolute measurement of the flows passing is not required. What is essential is that metering errors, if present, must be constant; once this is the case it is simple to apply a correction for the error on any given meter. The instrumentation in use on Nos. 1 to 4 superfractionators, as shown in Fig. 5, is set out in Table

IV and Figs. 10 and 11. In the case of No. 1 superfractionator, the original instrumentation provided for volume control, but during the commissioning period was changed over, on theoretical grounds, to temperature control, and this has proved completely successful.

TABLE IV.
Instrumentation—Isohexane and Isoheptane Production.

	Isohexane, Op. 1.	Isohexane, Op. 2.	Isoheptane, Op. 1.	Isoheptane, Op. 2.
Flows :				
Feed	F.R.C.	F.R.C.	F.R.C.	F.R.C.
Overhead	L.C.C. with F.M.	F.R.C.	F.R.C.	F.R.C.
Bottoms	L.L.C. sets F.R.C.	L.L.C. sets F.R.C.	L.L.C. sets F.R.C.	L.L.C. sets F.R.C.
Reflux	F.R.C.	L.L.C. with F.M.	L.L.C. with F.M.	L.L.C. with F.M.
Transfer	L.L.C. sets I.F.C.	L.L.C. sets I.F.C.	L.L.C. sets I.F.C.	L.L.C. sets I.F.C.
Steam to reboilers	I.T.C. (P. 13) with F.M.	F.R.C.	F.R.C.	F.R.C.
Water to condenser	F.R.C.	F.R.C.	F.R.C.	F.R.C.
Temperatures :				
Feed	I.T.C.	I.T.C.	I.T.C.	I.T.C.
Product	—	I.T.C.	I.T.C.	I.T.C.
Pressures :				
Column	I.P.C., plate 10 to vent.	Nil	Nil	Nil
Levels :				
Condenser, water inter- face	L.L.C.	L.L.C.	L.L.C.	L.L.C.

Notes : F.R.C. = Flow recorder controller.
L.L.C. = Liquid level controller.
F.M. = Flow meter.
I.F.C. = Indicating flow controller.
I.T.C. = Indicating temperature controller.
I.P.C. = Indicating pressure controller.

Referring to No. 1 Superfractionator (isohexane, Operation 1), the question of temperature and pressure control is discussed more fully in the following paragraphs.

Four methods are available for maintaining a constant pressure in this operation. First, consideration was given to the possibility of injecting air into the condensers, but, in view of possible fire risks, this was not given further attention. Secondly, the column pressure can be maintained by varying the supply of cooling water to the condenser, so that a constant vapour pressure is maintained in the system; this implies interconnecting the indicating pressure controller with the recording flow controller governing the water to the condenser, and suffers from the disadvantage that provision must be made for venting any non-condensable gases in the system. Thirdly, the temperature of the product, acting through a temperature controller, can be made to regulate the cooling water to the condenser by resetting the recording flow controller on the cooling water; in this case the indicating pressure controller acts merely as a safety valve for non-condensibles. Finally, and as actually adopted in Abadan, the unit

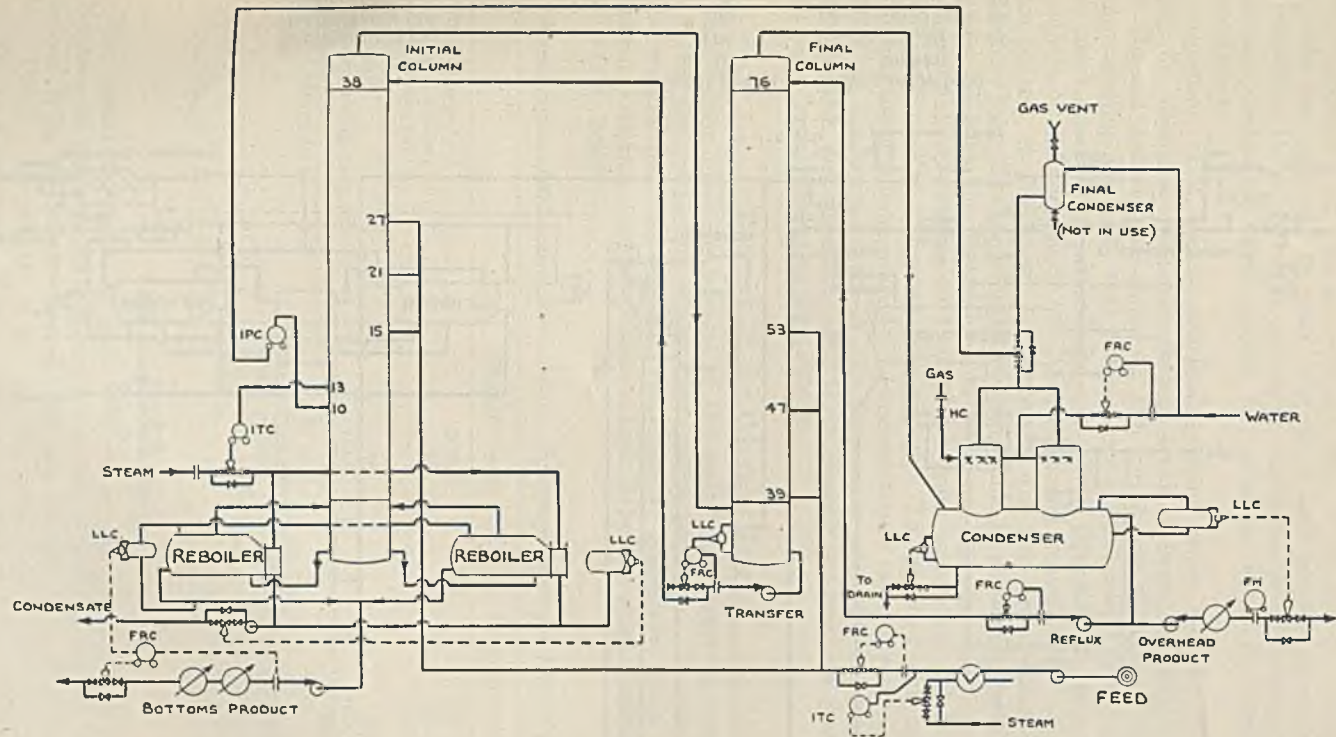
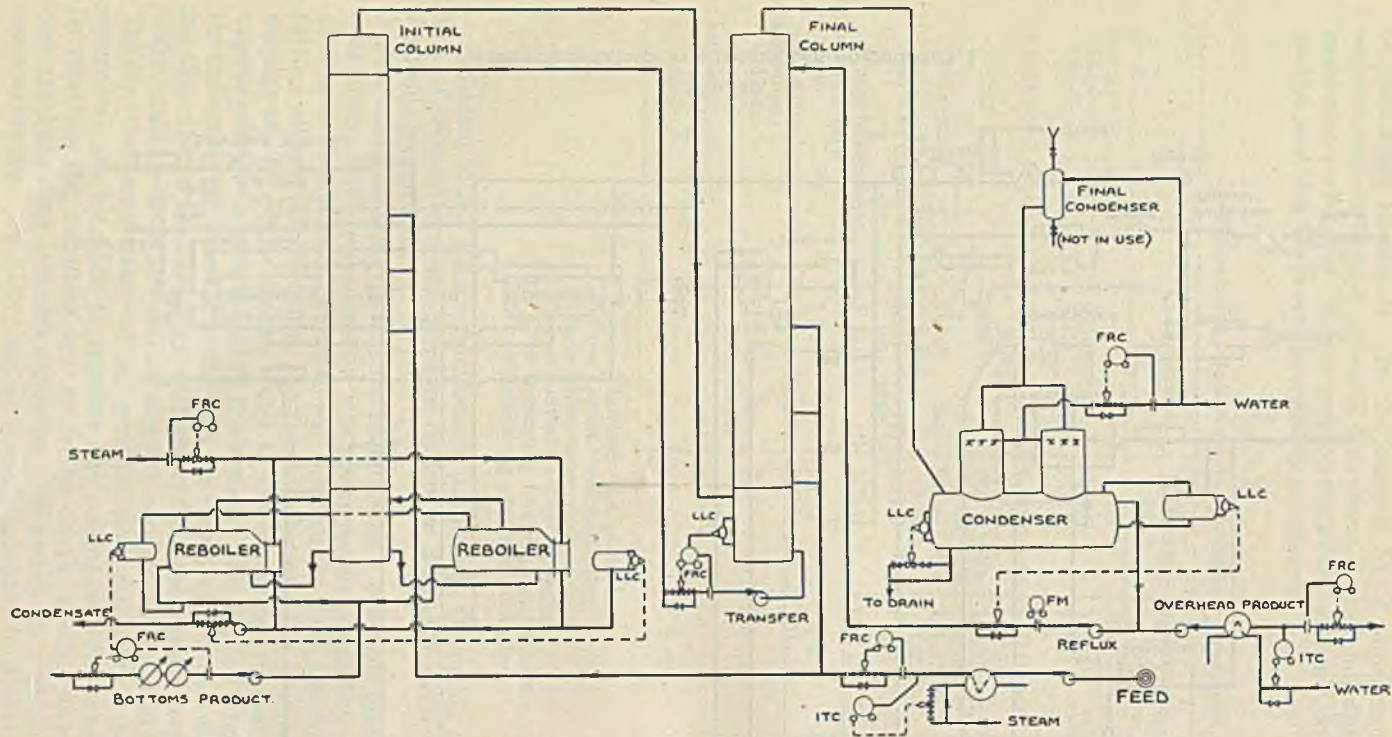


FIG. 10.
INSTRUMENTATION FOR ISOHEXANE—OPERATION 1.



	Total plates in unit.	Available feed plates.
Isohexane, Op. 2	100	19-27-35-51-61-69
Isoheptane, Op. 1	100	19-27-35-51-61-69
Isoheptane, Op. 2	76	15-21-27-39-47-53

FIG. 11.

INSTRUMENTATION FOR ISOHEXANE, OPERATION 2; ISOHEPTANE, OPERATION 1; HEPTANE, OPERATION 2.

can be pressured up with an inert gas such as flue gas, or, alternatively, H₂S-free fuel gas may be used. On No. 1 unit propane is continuously injected into the dome of the condenser, and is bled off after the indicating pressure controller; this method of operation has been found quite satisfactory.

Inspection of a typical true-boiling-point curve for the feed-stock (Fig. 6) shows a well-accentuated break between *n*-pentane (b.pt. 36° C.) and *cyclopentane* (b.pt. 49° C.), and it was thought, even before commissioning the unit, that temperature control would probably prove the most satisfactory method for controlling Operation 1 on isohexane production. In addition to the above theoretical reason for using temperature control, it was found, on processing the first and second tanks of isohexane base, that considerable layering could be encountered, due largely to the unavoidable fluctuations in both quality and quantity of the deisopentane gasoline and overhead from isoheptane, Operation 1, passing forward to isohexane base storage. To operate a unit on such a feedstock using volume control would necessitate as many true-boiling-point curves as there are layers in the base tank, and this is clearly both impracticable and unsound. For these reasons temperature control was employed in this operation with success.

ROUTINE PLANT CONTROL TESTS.

This section gives an account of the tests which must be carried out to ensure the production of constant-quality products. In carrying out these routine plant-control tests two types of laboratory batch still are required, and these should possess substantially the characteristics given in the table below:—

Characteristics of Laboratory Fractionation Equipment Used for Plant Control.

	40-plate batch still.	25-plate batch still.
Normal charge, ml.	2000	100
Theoretical plates	40	25
Static holdup, ml.	1	1.1
Total holdup at operating boilup rate, ml.	56	7.4
Operating boilup rate, ml.	1800	400
Holdup factor	2.7	2.6
Charge, holdup ratio	36 to 1	13 to 1
Packing	Fenske type single turn glass helices I.D. of 0.125 in. Wire diam. 0.02 in.	Spiral screen as used by Leckey and Ewell, <i>Ind. Eng. Chem.</i> , 1940, 12, 544.

Isohexane, Operation 1.

This operation makes a split between *n*-pentane and *cyclopentane*. To determine the amount of *n*-pentane in the bottoms product a sample is removed from the column and 2-litres are charged to a 40-plate batch-still. Using a reflux ratio of 40 to 1, the percentage distilling to 43 C. is reported as *n*-pentane. The amount permissible is naturally dependent on the percentage of *n*-pentane which can be tolerated in the final product.

Quoting from Abadan production, the *n*-pentane percentage as determined in the foregoing test varies from nil to 1.0 per cent. When commissioning a column on this operation, this test should be carried out at 4-hourly intervals, but once steady operation is reached a test every 24 hours is sufficient.

To determine the quantity of *cyclopentane* and *neohexane* leaving with the overhead—and so obtain the percentage recovery of these valuable components—the following test is made. One hundred ml. of the overhead sample is charged to a 25-plate still and the first 70 per cent. is distilled over at 20 to 1 reflux ratio. The distillation is continued at 40 to 1 reflux ratio and the fraction is collected between 80 per cent. and the minimum possible residue (about 10 per cent.). The residue is distilled to dryness to remove traces of grease, so that the final distillate now contains only *n*-pentane, *cyclopentane*, and *neohexane*. The refractive index of the 80–90 per cent. distillate and the redistilled residue are now determined, and the analysis is calculated from a knowledge of the refractive indices of the components by direct proportion, using values of 1.3931 for the refractive index of the *cyclopentane-neohexane* component, and 1.3576 for that of *n*-pentane. As in the case of the previous test, this estimation should be carried out frequently on commissioning the plant, but once this period is over a weekly determination will suffice, since with a substantially constant feedstock and operating at a given reflux ratio the recovery of *cyclopentane-neohexane* will remain unchanged.

Isohexane, Operation 2.

The bottoms product, from Operation 1, was allowed to accumulate in a 2,500,000-gal. tank. When this was full an analysis was made to determine its *cyclopentane-neohexane* content, and using the experimentally determined rich mixture blending factors, the exact location of the cut point was fixed for a given-quality product. The unit was then set to give the calculated percentage overhead, and a daily octane number provided a sufficient check on plant operation.

Isoheptane, Operation 1.

The function of the column engaged in this operation is to remove *n*-hexane overhead with the minimum quantity of benzene-methyl*cyclopentane* components. The specification employed limits the amount of *n*-hexane in the final isoheptane, and in order to determine and control this quantity, the following control test has been developed. Two litres of bottoms from the unit are charged to the 40-plate batch still in the laboratory; heat is applied, and the column allowed to reach a state of equilibrium under total reflux. The column-head temperature is recorded at the end of half to three-quarters of an hour. Experience has shown that if this figure is 70° C., the final product will be satisfactory. If, however, it is as low as 69° C., the *n*-hexane content of the final product will exceed that allowed by the specification (4 per cent. vol.), while, on the other hand, a figure of 71° C. would indicate an excessive loss of benzene-methyl*cyclopentane* in the overhead. This test is carried out every 8 hours, and, in spite of its empirical nature, has been found completely satisfactory. Dependent on the temperature registered, an alteration is

then made to the overhead meter on the unit, a figure of 71° C. would result in the overhead percentage being reduced by 1 to 1½ per cent. based on the feed. The correct setting of this unit is also aided by the analysis of each tank of naphtha feed, but the final adjustments are always made using the test as just described.

Isoheptane, Operation 2.

The function of this operation is to remove overhead isoheptane containing only a small amount of material boiling above 95° C., and it has already been indicated that the cut point when producing a specification product lies on a flat. Based on experience, and with a knowledge of the analysis of the naphtha feedstock used for the previous operation, this separation can be carried out with no plant control other than gravity determinations on the overhead at 4- or 8-hourly periods. On the result of these determinations small alterations (± 2 per cent. on the feed to the unit) are made to the overhead product meter so as to give a constant gravity product.

If for any reason it is required to produce a lower-grade product containing a higher percentage of methylhexanes, it is necessary to take a deeper cut on this operation, so that the cut point lies nearer to 95° C., and the operation is more critical. In this case the gravity of the overhead product is of less significance, and the percentage of inferior material boiling at the back end can only be determined by a full distillation as described earlier, or by spectroscopic means as outlined in the following paragraph.

This section would be incomplete without mention of the use of ultra-violet spectroscopy in the analysis of isohexane and isoheptane. It will be recalled that in isohexane, Operation 2, *n*-hexane is being separated from the 2- and 3-methylpentanes, and the final product should contain a minimum of the hexane diluent. On account of the peculiar vapour-liquid relationship existing between *n*-hexane and benzene, the former of these two components always contains traces of the latter, and the practical application of this fact constitutes a rapid method for estimating the amount of *n*-hexane in a sample of isohexane, and one which can readily be used as a plant control test for isohexane, Operation 2. In a similar manner our specification for isoheptane sets a limit of 5 per cent. for the amount of material boiling above 95° C. on the true-boiling-point distillation, and this material contains traces of toluene. For the ranges with which we are concerned the ratio of the toluene concentration to the non-aromatic portion of the material boiling above 95° C. shows little variation, and again, using the ultra-violet instrument, a speedy control test for isoheptane, Operation 2 can be developed.

ACKNOWLEDGMENTS.

The authors wish to emphasize that the work described represents the combined efforts of a considerable number of the technical personnel in the various departments of the Company; they would like to take this opportunity of acknowledging the important part which they played in bringing the project to a successful conclusion. They also wish to thank the Chairman of the Anglo-Iranian Oil Company for permission to publish this paper.

THE INSTITUTE OF PETROLEUM.

A MEETING of the Institute of Petroleum was held at 26, Portland Place, London, W. 1. on Wednesday, January 9, 1946, the President (PROFESSOR F. H. GARNER, O.B.E.) in the Chair.

A paper entitled "Process Development and Production of Isohexane and Isoheptane as Aviation Fuel Components," by S. F. Birch, P. Docksey, and J. H. Dove [see pp. 167-199], was presented by Mr Docksey.

MR. DOCKSEY, introducing the paper, said the paper was an attempt to describe the development and final production of two components of the 100-octane spirit which was manufactured in Abadan. These components were in the nature of a base stock. They have been called "isohexane" and "isoheptane," not using those terms in the chemical sense—i.e., as the organic chemist would use them—but simply as representing concentrations of hydrocarbons from which the normal paraffins have been excluded. There is no intention of using the terms in this paper to indicate the pure compounds which were normally referred to in that way.

DISCUSSION.

THE PRESIDENT said the remarks of Mr. Docksey would indicate the vast amount of work involved in the developments described in the paper. One could regard as the beginning of this development the fractionation work done by Sydney Young early in this century, when he developed the evaporator still head.

Many would remember the statements made about ten years ago concerning the ideal process of fractionating petrol so as to take out heptane and other low-octane number fractions to give a good anti-knock quality petrol. At that time doubt was expressed about its accomplishment within a reasonably short time; but as would be seen from the paper that all that, and very much more, had been accomplished by special fractionating methods.

The true-boiling-point analyses were reported by plotting the weight per cent distilled against the temperature of distillation. That was the normal method of reporting these true-boiling-point analyses, both in Great Britain and in America. He always felt that if the *rate of distillation* was plotted, rather than the actual percentage off, it was very much easier to follow where the peaks occur and also the efficiency of separation achieved. But, of course, that had the disadvantage of requiring a planimeter to estimate the yield to the various points.

MR. DOCKSEY agreed entirely with the President concerning the usefulness of the differential curves. They called them the composition curves, curves which were really obtained either by differentiating the true-boiling-point curves as given in the paper, or else by splitting them into a number of arbitrary fractions, say 1 per cent fractions, and dealing separately with

each fraction. They gave a large amount of information and, to anybody interested in distillation, were vital. That way of looking at true-boiling-point curves was well worth cultivating. He had not seen it fully described and thought it would be a good thing if it was described at some time.

THE PRESIDENT said it was described by Young.

MR. DOCKSEY said that was so, but it had not been used subsequently so much as he thought it might have been. It could be used with advantage with the higher-boiling-point materials also, where one got right into the lubricating-oil range.

DR. K. E. W. RIDLER was very interested to hear the references to quality control by remote-control methods, and asked for some idea of the energy which had to be wasted by way of safety factors in covering the control at Abadan?

MR. W. A. PARTRIDGE (who was invited by Mr. Docksey to reply to the question), said that on isohexane and isoheptane no safety factor was allowed, but a safety factor had been applied to the finished blends in the early days at Abadan. A number of blends covering possible variations in component production rates were prepared at Sunbury and approved by the M.A.P. for production in Abadan. Then, those responsible at Abadan, chose any composition or combination of compositions to suit the component production rates. A safety factor, which was not very large, was applied on each blend at Sunbury; the safety factor allowed on a rich mixture amounted to about 1 per cent. So far as he was aware, there was no failure to produce specification fuels at Abadan.

DR. W. R. VAN WIJK referred to Fig. 10 and said that on the left there was an indicating pressure controller. He asked what was the control there and was it meant to blow-off gas? *

MR. DOCKSEY said that was described in the paper. This column was operated under pressure for condensing normal pentane and asked Mr. Dove, who had seen most of the operation of the pressure control on this column, to deal with the question.

MR. J. H. DOVE said that on the right-hand side of Fig. 10 there was a gas inlet at the top of the jet condenser; gas was continually available at that point, and the indicating pressure controller referred to, on the left-hand side of the figure, was set at the pre-determined pressure of 30 to 40 lb, and kept the whole unit under that pressure.

DR. VAN WIJK asked whether it was outside gas?

* The line in which the pressure controller is assembled is drawn as a transportation line. It is now understood that it is actually a control line only. The question arose from the fact that the line was considered to be meant for delivery of gas from the column to the condenser or reversely, for which no reason could be seen.

MR. DOVE said it was outside gas; mainly propane obtained from elsewhere.

MR. C. R. FRANKLIN, referring to the temperature controller on the column, said he thought it had been stated that there was difficulty in controlling the reflux according to the temperature at any particular plate on the column, and that the volume control method had been used instead. He asked what was the objection to using a ratio controller for the reflux on such a column, which could be set according to the quality of the feed coming in?

MR. DOCKSEY said he had not intended to give that impression. There was no difficulty in controlling the reflux by a temperature controller; but what happened when that was done was the production of a very variable product composition when one was trying to separate such close-boiling materials. The second part of the question, he presumed, was whether a ratio splitter on the reflux could have been used instead of volume control. That, of course, was also controlling on volumes; it was not controlling on temperature. But the complication involved had to be considered: it was controlling on volumes, and if that was done it was necessary to have a constant proportion of the feed coming out as distillate. A constant amount of feed going in could be maintained and, therefore, if the overhead was to be split in a constant ratio it must be somehow ensured that the weight of vapour going overhead into the condenser, and so into the reflux drum, was constant; otherwise there would not be a constant amount of distillate withdrawn from the column. On the whole, it was more difficult to ensure that than to use the method adopted in Fig. 11, although he saw no objection to it theoretically; it was more difficult to ensure that a constant amount of distillate was withdrawn.

To take a standard case, and reducing the problem to a small scale, supposing one had a column into which 100 gallons per hour was fed, and it was known that to withdraw 30 gallons of distillate per hour constantly would give the right quality. Supposing there was a reflux ratio of 10:1; that meant that there would be 300 gallons per hour falling from the top plate, and the equivalent weight of vapour going up. Supposing also that the heat lost during the day would condense 10 gallons out of the 300, but that at night, when the temperature generally was lower, it would condense 30 gallons. The net amount going overhead was 330 gallons during the day, but obviously it would be only 310 at night. So the ratio would have to be altered in order to maintain the 30 gallons per hour coming out. Thus, it was theoretically possible, but not so easily applicable in practice as was the other method.

MR. E. THORNTON said he was delighted to see this method of volume control coming into its own, because it had been used in Llandarcy for some years and, from an operators' point of view, was regarded as almost foolproof. Since it demanded no theoretical knowledge of why it functions and was capable of giving maximum efficiency of operation, they had long thought it had merits over control by observation of physical data like temperature and pressure. Reflux and heat input could be adjusted without

reference to temperature, pressure, etc., to maintain quality within the capacity of the column to any desired standard on a fixed quantity, corresponding to analytically ascertained available content in the feed.

The method was, of course, entirely dependent on a constant quality of stock being available over long periods and on accurate analytical assay of such stock, but, given these two requisite conditions, the method was better than any other that he knew for making sure that the available content of a particular fraction was obtained as well fractionated as the characteristics of the column with reference to the throughput permitted, and with the minimum necessary reflux, since this could be reduced under observation from some excessive amount to the minimum which would still maintain quality on the correct quantity.

He took the opportunity of paying his tribute to the daring imagination and the persistence of all concerned in obtaining this essentially laboratory standard of accuracy on a full-scale works' production—a very fine piece of work and very excellently reported.

MR. PARTRIDGE emphasized that this method of making an aviation base stock was very flexible. It would be observed from the rich mixture ratings of the various components, with both isohexane and isoheptane, that there was very considerable difference between the front fractions and the later fractions in both cases. This meant that it was possible, within a very short space of time, to alter the quality of the isohexane or isoheptane in an upward direction, although it was true a sacrifice in yield was necessary. So that if at any time it was desired to make an aviation fuel of quality higher than that at present required, a base stock for making that higher quality fuel could be produced by alteration of plant conditions.

MR. K. B. ROSS referred to a volume control which had been worked out for his firm, and which had been used very effectively to start the fractionators. The last time he went around the units he found the volume control being used as a guide rather than as a method of control, and that the operators had reverted to the old scheme of working on gravity; they found it was satisfactory, if they used the volume control as a guide to start them off, so that super-fractionation had become a fairly easy operation.

MR. DOCKSEY did not agree, and considered it a confusion of the mind between the specification it was desired to control and the method by which the actual controlling was performed. In a case where the gravity was controlled, and supposing the gravity was wrong, what the operator did, he presumed, was to notch up or down the flow controller on the distillate offtake?

MR. ROSS said that was so.

MR. DOCKSEY said that was volume control.

MR. ROSS agreed but said it was only as a guide.

MR. DOCKSEY said that was not so, it was the control of the plant. Whether or not the operator notched it up 1 per cent because the gravity

was low, or because somebody from the laboratory came along and said that he was going on to a new tank and it would have 1 per cent more than he had had on the last one, was a matter of no importance, it seemed to him. He thought one should distinguish between the specification to which one controlled and the mechanics of actually doing it. If it was required to control gravity and a recording gravity meter was installed—an instrument which he understood it was possible to obtain—and that was made to operate the reflux return, then it was no longer volume control; it was getting quite close to gravity control. But the mechanics Mr. Ross explained was still really volume control, using gravity as a guide.

MR. G. BAARS, comparing Figs. 10 and 11, said that in the one case there was the normal cooler in the top product, and in the other case there was a heater in the top product. Was that done specially by reason of the flow control, to ensure that the temperature was the same always? Which was the better control, that in Fig. 10 or that in Fig. 11, or were they equally effective?

MR. DOVE asked whether Mr. Baars was referring to the cooler on the overhead product in Fig. 10, and to the heater in Fig. 11, which latter was marked "I.T.C."?

MR. BAARS asked whether those arrangements made were made specially to control the quantity?

MR. DOVE said that in the initial stages they were very worried about metering, and there was a slight misunderstanding, in so far as the erroneous idea got about that they required absolute precision in metering. That was not quite what was wanted; rather did they want constant metering. In other words, it really did not matter if a meter showed that $34\frac{1}{2}$ per cent overhead was being taken when the real figure was 34 per cent; but it was necessary to know that if the meter read $34\frac{1}{2}$ per cent one day it should read $34\frac{1}{2}$ per cent the next day. So the temperature of the streams going into the meters was controlled and that accounted for the heater in Fig. 11. The product coming out was maintained at a constant temperature by the indicating temperature controller.

MR. BAARS asked if it was a cooler in Fig. 10 and a heater in Fig. 11?

MR. DOVE asked Mr. Ross to deal with that point.

MR. ROSS said surely the cooler in Fig. 10 ensured that the distillate went forward to storage adequately cooled, no particular provision being made to control it to any temperature. In Fig. 11, however, it was purely a matter of temperature control to ensure that the orifice plates received the material at a constant temperature.

MR. E. THORNTON, in a written contribution, said "The controversy in the discussion between Messrs. Docksey and Ross does not clearly bring out the real merit of this system of volume control as defined earlier in the paper, and as I understand it.

"I accept the definition given in the paper which reads, 'The principle on which volume control is based is the control of the quantity of one of the product-streams withdrawn from the column as a fixed proportion of the feed, the quantity of the product so withdrawn being determined by a true boiling point analysis of the feed.'

"This is something rather different from the mere use of a flow meter instead of a thermometer or a gravity meter, as suggested by Mr. Docksey in his comment on Mr. Ross's observation that the plant operators were tending to go back to controlling the specific gravity, using the volume-control method merely as a guide. Always on the assumption that the feed was constant and the percentage content had been accurately determined by analysis, once the column was set to take off a fixed percentage of the feed, a variation in quality as indicated by a change in gravity, refractive index, or any other property, should be assumed to be a breakdown in the column functioning as a fractionator—*e.g.*, insufficient reflux/heat input balance—and the correction of quality should be sought in the first place by adjusting the heat input and reflux in an upward direction, and not by reducing the quantity taken off. Admittedly the metering may have gone wrong, in which case a re-setting would be necessary, but this could be checked by dip over a period. The danger of reducing the quantity of offtake to correct inferior quality would be that it would be accepting an inefficient or over-loaded column and would inevitably result in a failure to obtain all the available content of the fraction desired in the offtake.

"I cannot agree with Mr. Docksey in his remarks, and would say that the essence of this method of control comes in putting the emphasis on 'fixed' rather than on 'volume' and adjusting everything that is possible by any means at one's disposal to maintain quality in this fixed quantity. If such correction is beyond the power of the operator, than it means that, for some reason, such as excessive input or chokage of the column, the equipment is no longer capable of giving this maximum yield, and should be shut down when the reduced input becomes uneconomic."

Vote of Thanks.

THE PRESIDENT, proposing the hearty thanks of the Institute to the authors, said it would be agreed that the paper would rank amongst the highest published in our Journal of the Institute. There was so much matter in it that it needed careful study.

It only remains for him to ask the meeting to accord a most hearty vote of thanks to the authors and to all who were associated with them in the work described.

(The vote of thanks was accorded with acclamation.)

THE MECHANICAL TESTING OF EXTREME PRESSURE LUBRICANTS (WITH SPECIAL REFERENCE TO HYPOID OILS).*

By H. L. WEST.

INTRODUCTION.

THIS paper has been prepared at the request of the Mechanical Tests for Lubricants Panel of Sub-Committee No. 5 of the Standardization Committee of the Institute of Petroleum.

In view of the increasing use of axles requiring E.P. lubricants, the question of adopting for standardization one or more of the mechanical tests which have been proposed for testing such lubricants was considered. There exists a variety of machines, and tests on these machines, none of which has yet been universally accepted. The question, therefore, of adopting any particular method of test on any one machine with a view to making it a standard method could not be pursued. On the other hand, a number of specifications are in existence which call for tests on one or other of these machines. It was decided, therefore, that a paper should be prepared which, besides giving details of the test procedures and research work on the five most-used machines, should also give the specification tests and the specifications which call for these particular tests: this latter information is given in the Appendix. By presenting the information in this way it was hoped to fulfil two purposes: (a) to have a source of information on the subject in a readily available form; and (b) to promote discussion, interest and progress in this complicated subject.

HISTORICAL.

Miller, in the General Discussion on Lubrication and Lubricants organized by the Institute of Mechanical Engineers made the statement that:—

“Perhaps no recent single development in the field of lubrication has done more to revolutionize thought on lubrication than the introduction of the hypoid gear.”¹ This statement whilst undoubtedly true, should be coupled with another, *e.g.*, Tuplin—namely, that “it appeared that the hypoid gear held the doubtful distinction of having led to the development of E.P. lubricants because, as an automobile rear axle drive, it would not work satisfactorily without them.”² Which ever view is taken, it is beyond doubt that the hypoid gear has accelerated what was, perhaps, an inevitable development.

In explanation of the need of a special lubricant for the hypoid gear, Dahlstrom³ has pointed out that a much greater pressure can be applied to gear-teeth than to bearings because of the much shorter time of contact and the much smaller relative sliding motion. If the latter is increased, as in the hypoid gear, either the unit pressure must be decreased or an

* Paper read before a meeting of the Institute of Petroleum, January 27, 1944.

E.P. lubricant used to prevent scuffing; this in spite of the fact that the surface pressure on the hypoid is less than on a spiral bevel gear of comparable dimensions.² It is the combination of high surface pressure and sliding velocity of steel on steel that demands the special type of lubricant. Gears can be operated successfully with these special lubricants, whereas scoring and failure are almost certain to occur with ordinary mineral gear oils. They are not only required by the hypoid gear, but by highly loaded spiral bevel and industrial gears.

The development of the various machines for testing E.P. lubricants has followed closely the development of the hypoid gear itself. According to Griswold⁴ the hypoid gear was first made by the Gleason Corporation about 1924 and was adopted by Packard about 1926. For many years this was the only car with the gear and, since it was a premium-priced car, and would be serviced by Packard service stations, not much difficulty was experienced. The advantages of the gear in giving reduced gear noise and a lower floor level led to its more widespread use. By 1935 11 per cent. of the cars produced in the United States carried E.P. lubricant recommendations; from this time there was a rapid rise in such recommendations. Based on experience with cutting oils, etc., the early lubricants contained sulphur and lead soap, this type being in use in 1931. Other "milder" gear oils were produced, but the majority of these were unsatisfactory in the hypoid gear, and up to about 1939 the lead soap—"active" sulphur type—held the field almost exclusively. An alteration of outlook on the lubrication of hypoid gears followed the introduction of these axles into trucks. For the latter it was found that the "active" types were unsatisfactory, causing undue wear at high loads at low speeds, whilst some of the so-called "mild" E.P. oils were satisfactory. Two different types of lubricant were then suggested, a passenger car-duty and a truck-duty hypoid lubricant.⁵ Since that time a universal lubricant has become available incorporating both qualities, and to-day all Military Service vehicles are being lubricated with such lubricants. Coupled with the increase in the use of the hypoid gear has come the use of much higher tooth pressures due to the reduction in the size of the gear to transmit a given horse-power.

As is only to be expected, much of the development work on hypoid lubricants and methods of testing them has been carried out by the gear manufacturers. For this purpose two test procedures have been evolved, one type using a car, called the "Shock Test," and the so-called "Four-Square" axle rig. The former method has been described by Prutton and Willey⁷ and the latter by Prutton and Willey and Witham.⁸ For completeness the details of the "Shock" or "Bump" test and the dynamometer test are given in the Appendix. Since full-scale tests are costly, time-consuming, and tedious, it is not surprising that the gear manufacturers have interested themselves in the development of laboratory machines as exemplified by the large General Motors, the Almen, and the Timken machines, as well as co-operating in the design of the S.A.E. machine. Oil companies and additive manufacturers have also, naturally, been active in developing test machines; these are exemplified by the Floyd⁴⁷ the Four Ball, the Brownsdon¹⁰ and the Smith Wear tester⁴⁸ and many others, details of which have not been published.

LABORATORY TESTING MACHINES.

It is proposed now to discuss each of the more widely used machines—namely, the Almen, Timken, Four-Ball, Falex, and the S.A.E., describing their modes of use, advantages, and limitations as disclosed in the published literature. In certain instances this can be supplemented by some unpublished observations which the writer has collected.

The Almen Machine.

The large "General Motors" machine¹¹ was first described in 1931 and, from the data published, appeared to correlate reasonably with service. Experience showed, however, that this correlation was not so good as at first expected and, coupled with the desire for a less expensive and less cumbersome machine, led to the development of the Almen machine.

The description of the machine and the normal method of operation are given in the Appendix. Wolf and Mougey¹² have described the machine and given results obtained on a large variety of E.P. oils, tests using various journal speeds being recorded, the effect being that at the lower speeds the test was less discriminating. Photographs were included showing the condition of the journal after each test. Data were also presented showing the correlation with service tests in cars and in the Gleason modified "Four-Square" rig. The conclusions drawn from the results were that, although correlation was not perfect, the Almen machine did show which lubricants were likely to behave well in service.

This view was modified later when further experience was gained. Neely¹³ reviewing an investigation carried out at the Bureau of Standards,¹⁴ showed that variations in the test procedure, including the use of different metals, rate of loading, and speed, failed to improve the correlation. A comparison was also given of results obtained with the Almen and large General Motors machines and a worm-gear test. It was concluded that the machine, whatever the operating conditions, was not capable of rating lubricants according to service. Comparative tests with a variety of service experience have been given by Prutton and Willey¹⁷ and James,¹⁶ the latter quoting some of the results of Prutton and Willey. Evans^{17, 18} has described the use of the machine to measure the film rupture strength of oils and greases to which various additives have been added. The results were expressed as pounds per square inch load at seizure.

The Almen machine, like many others, showed—at first—considerable promise as a measure of the service performance of extreme-pressure lubricants. The design of the test-pieces would appear to make it difficult to be sure whether fluid or boundary conditions or a combination of the two are being employed. The motion is purely one of rubbing of one metal on another, there being no rolling motion similar to that of a gear. Furthermore, the rubbing speed is low, there is no temperature control, and the metal used for the test-pieces is not similar to that used in gears. It would appear that its use has now been confined to testing some mild E.P. and cutting oils.

The Timken Machine.

The first published description of this machine was by Wooler¹⁹ in 1932, who stated that it was developed because of the inability to obtain the desired information from the oil industry. It has the advantage over the Almen machine of allowing a fairly accurate friction measurement to be made; it was also stated that the temperature rise follows the friction curve. Since the scar width can be measured as well as the test-pieces being weighed, abrasion tests can be carried out; from such tests it was concluded that sulphur caused abrasion. Lincoln, Byrkit, and Steiner²⁰ have used the machine in an investigation of the effect of organic chlorine compounds on the Timken film strength, and suggested an "index" to express the effective improvement obtained. They also described the use of a modification, which, although it is believed this is the only published reference, has been widely adopted. Instead of applying the load instantaneously, the weights are supported by either a hydraulic jack or a motor-driven screw-jack. To apply the load the jack is lowered under specific conditions, which can be repeated at will. Although this application of the load is, to all intents and purposes, instantaneous, it does influence the results obtained. Evans,²¹ who has given a detailed description of the machine and its method of use for both oils and greases, has reported results which show that sulphurized oils were more "abrasive" than the chlorinated variety. Prutton and Willey⁷ have recorded tests using oils on which they had also carried out full-scale axle tests; little correlation between the two could be found.

In his preliminary investigation, Wooler¹⁹ studied the effect of the speed of rotation of the cup on the results obtained, the speed being varied from 200 to 1000 ft/min. As only three oils were tested, the results were not very significant. McKee and his co-workers¹⁴ examined the effect of speed and also temperature on the load-carrying capacity, and confirmed Wooler's results, the effect being to decrease the load carried as speed and temperature were increased. Southcombe²³ has recorded unconventional tests using the Timken machine rather as a research instrument than as a "go" and "no-go" machine, and showed that the actual intensity of the load may bear little, if any, relation to the load applied. The effect of the speed of rotation was also emphasized, the effect varying with the lubricant; temperature, on the other hand, was said to have little effect. Van der Minne²⁴ has shown that it is possible, at speeds as high as 2000 r.p.m. (1000 ft/min), to reverse completely the rating at low speeds and, in general, the E.P. oils showed the best behaviour at the higher speeds. The curves in Fig. 1 taken from this paper illustrate this point.

Neely,¹³ reporting the results of McKee,¹⁴ observed also that when the machine was mounted on a wooden table, the results differed from those obtained when it was mounted on an iron stand, the effect being due to the rigidity of the mounting. A comparison was also given of the results obtained with a worm-gear machine, and those obtained with the Timken machine. For estimating the abrasive character of E.P. lubricants Miller¹ has used a modification of the machine, a ball-bearing being employed instead of the test-cup.

Evans²¹ has shown that the smoother the finish of the test-pieces the greater the load carried and the lower the co-efficient of friction. Baxter,²² investigating the effect of surface finish and pre-treatment of the test-cups, has demonstrated that pre-treatment with E.P. dopes, and even hydrochloric acid, can materially alter the load carried even with undoped mineral oils.

It has been reported by Maag²⁵ that the viscosity influences the results obtained with the Timken machine, and Southcombe²³ has confirmed this, at least for certain temperatures. This indicates that the conditions are a mixture of fluid and boundary lubrication depending on the load, temperature, and speed at which the machine is operated. The motion of the machine is pure sliding, with no rolling action.

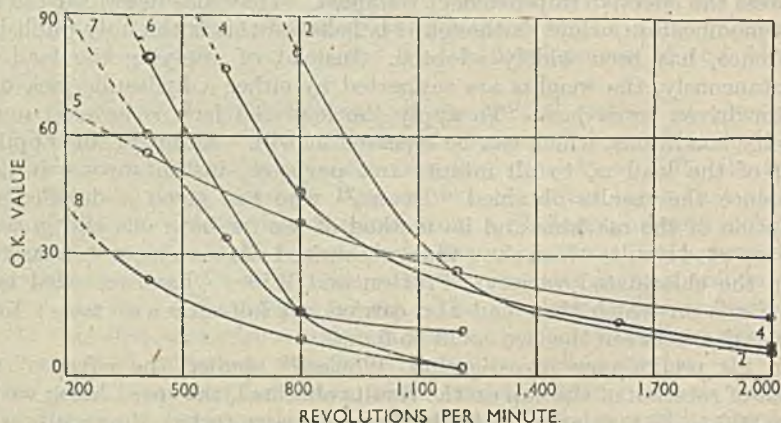


Fig. 1.

RELATION BETWEEN SPEED OF ROTATION AND "O.K. VALUE" OF THE TIMKEN LUBRICANT TESTER (24).

It should be pointed out that the standard test-cups and blocks used in the U.S.A. are fabricated from a different alloy from that used by British Timken—namely, in the U.S.A. a nickel-molybdenum steel, and in the U.K. a chrome steel is used. This may vitiate comparison of the results obtained in the two countries; furthermore, in the U.K. the test-cups are reground for further use until they reach a minimum diameter. This is discussed more fully under the S.A.E. machine. This situation is liable to be altered should the two companies alter their raw materials.

The machine has been described in two British journals.^{26, 27}

The Four-Ball Machine.

Most reported investigations using the Four-Ball machine have used the one-minute procedure first described by Boerlage²⁸ and given in full in the Appendix. Results using this method have been reported by Van Dijck and Blok²⁹ and Clayton.^{30, 31, 32, 33} The lubricants studied have ranged from petrol, mineral oil, fatty oils, to E.P. lubricants, and even glycerine and syrup. The observations made are usually the seizure, the recovery from seizure, friction, and wear; sometimes seizure would take place only after a lapse of some seconds. Clayton claimed an accuracy

of ± 5 kg and stated that no significant difference had been observed in different consignments of balls used in the test; the method of cleaning was also not critical. It was found that E.P. oils, in general, would carry higher loads without seizure, and that the recovery from seizure was quicker than with straight mineral oils; however, the wear at low loads was greater. Van Dijk and Blok have compared and discussed the test conditions with those of the other popular machines.

Clayton³⁴ has reported experiments in which the test conditions have been altered from that given above. Normally, the load is applied before switching on the drive. It was found that starting the machine before applying the load did not alter the results; therefore the original procedure was used throughout. By continuously increasing the load it was found that the seizure load was approximately 40 per cent higher, and that, within the range of the tests, the rate of loading did not influence the results; the scatter of the readings, however, was disappointing. As expected, an increase in the running speed gave lower seizing loads and shorter times to seizure; the machine vibrated badly at higher speed, and there was a wide scatter of the readings. Some observations were also given on the rigidity of the ball-chuck mounting and its effect on the results obtained with the machine. Blok,³⁵ in an effort to improve the correlation with service results, has proposed the use of the $2\frac{1}{2}$ -sec seizure delay method. In this, the machine was run, and only the first $2\frac{1}{2}$ sec were considered at each load. If no seizure was indicated on the chart in $2\frac{1}{2}$ sec, the load was increased until it was indicated. Results were given which showed that in comparison with the S.A.E. and Timken machines the $2\frac{1}{2}$ -sec seizure delay method gave much better correlation with high-speed hypoid operation. The reason for this is claimed to be due to the effects of bulk heating, which, it is suggested, should either be eliminated or controlled. In this test it is said that the temperature does not rise above ambient; Clayton³² has suggested an even shorter time of operation. Blok stated that some satisfactory E.P. oils showed a mild seizure at low loads with low wear. This should be disregarded, and if the load was increased, a true seizure would be obtained. Kemmerer and Garton³⁶ have suggested a method of rating truck hypoid oils by running tests for 5 minutes at 100-kg load and 1500 r.p.m., the wear-spot diameter being measured and indicating the suitability of the oil. It was stated, however, that further work was in progress to establish a correlation more definitely.

Clayton³³ has also experimented with test-pieces of different materials, tests being made with all bronze balls, bronze on steel, and steel on bronze. The results were different from previous tests with steel, due partly to the lower yield value of the bronze. With a single bronze ball on steel quite different behaviour was found, and the investigation was not pursued. With steel on bronze much higher loads could be carried even by kerosine; the possibility of film formation due to yielding of the bronze was suggested. Baxter, Snow, and Pierce²² have reported investigations on the use of pretreated balls, the pretreatment being with chlorinated or sulphurized dopes, or even hydrochloric acid. The results showed that higher loads could be carried with mineral oil and pretreated balls, but the load carried after seizure was unaltered.

The advantages claimed for this machine are the readily available test-pieces, and the absence of fluid film lubrication; also high contact loads can be readily obtained without the use of unduly heavy weights. The absence of fluid lubrication eliminates any viscosity effect. On the other hand, the rubbing speed is low and there is no rolling motion. It is to be assumed that the investigations in the various countries have all used S.K.F. balls, which are said to be of chrome steel.

The Falex (Faville-Levally or Cornell) Machine.

This machine does not appear to have gained very wide acceptance as a test machine for rating E.P. oils. Invented by Cornell,³⁷ and assigned to the Faville-Levally Corporation, many different test procedures have been suggested, including that patented by Faville.³⁸ This method, which is recommended for E.P. oils, involves testing the oil at increments of load, each being held for 1 minute, and noting any abrupt increase in torque, indicating incipient seizure. At 1500-lb load a 3-minute run is allowed. If no seizure is observed, the oil is diluted with straight mineral oil of approximately the same viscosity and the percentage of diluent found at which a failure at 1500 lb is just obtained; the test is commenced with the oil heated to 210° F. This procedure is open to criticism because Neely¹³ has shown that with some additives the lower concentrations may adversely effect wear, whereas higher concentrations arrest it. Another usual test is that described by Baxter and his co-workers,²² in which the load, after initial running-in, is increased continuously and the torque increases observed. Breakdown is indicated by a rapid rise of the torque or shearing of the brass pin, or even of the hole in the journal. The test method given in the Appendix is really a wear test, and is a modification of that originally laid down, in which the wear was measured during a period of 1 hour. It is worth noting that with this latter method this machine, in common with all the "sliding" machines, gives a high rate of wear for the "active" sulphur types of lubricant and, in general, low wear for the chlorine addition type.

Baxter²² has investigated the effect of pretreatment of the test-pieces, and no difference could be observed from the untreated pin. This was thought to be due to the softness of the pins and blocks, but on hardening the metal it was found that the breakdown loads were beyond the limit of the machine. It has been the writer's experience that the test-pieces for this machine are very variable, not only in hardness, but in the angle of the V and in finish. Using a recent delivery of pieces and the method of test given in the Appendix, it was found that a wear of approximately ten teeth may be obtained with certain oils. The previous supply of test-pieces when used for testing the same oil gave a wear figure of anything from sixty to 100 teeth.

Prutton and Willey⁷ and James¹⁶ have given a comparison of tests on the Falex machine and full-scale axle tests and showed that the correlation was poor.

The S.A.E. Machine.

This machine was developed as a co-operative effort of the gear manufacturers and the oil industry specifically for testing oils for rear-axle

drives. Its design appears to be based on the experimental machine used by the U.S. Bureau of Standards. Results obtained with this latter machine have been described by McKee, Bitner, and McKee.³⁹ In 1935 the S.A.E. Sub-Committee⁴⁰ suggested that a hypoid oil should carry 550 lb, and a milder type for spiral bevel gears 250 lb at 750 r.p.m.: the machine was, however, to be modified in detail. Neely¹³ in 1936 gave the first published description of the machine.

The novelty of the machine is that both test-pieces revolve, so that the applied load per unit area is not decreased by wear, but only by deformation at the point of contact. It is possible to vary the ratio of rolling to sliding within certain defined limits, and presumably the speed of the mainshaft could be varied at will. In this manner the action of gear-teeth in mesh is simulated. The test conditions usually used for rating hypoid oils at the present time are 1000 r.p.m., 14.6 : 1 rubbing ratio, and 83.5 lb/sec loading rate (*i.e.*, 8.35 lb/sec on the scale).

Results obtained on a variety of oils using this procedure have been published by Miller,¹ Zwahl,⁴¹ van der Minne,²⁴ Prutton and Willey,^{7, 15} and James.¹⁶ Miller stated that it was fairly agreed that the S.A.E. machine under the above conditions showed the most consistent correlation with practice. Zwahl outlined the Chevrolet Company's procedure in testing lubricants, and stated that it does not follow that an oil which failed on the S.A.E. test (*i.e.*, 550 lb) necessarily failed in practice. "It is our opinion that the S.A.E. machine results considered alone cannot be used as a basis for making a decision to approve or not to approve hypoid lubricants." Test results, combined with chemical analysis for active ingredients, coupled with experience, did, however, enable some prediction to be made. Prutton and Willey, and James, compared the S.A.E. results with tests made under a variety of conditions on the four-square rig, together with the Chevrolet and Packard shock tests, and showed that the shock tests require a high load to be carried on the machine, but that high-torque, low-speed tests on the four-square rig required other factors. Blok³⁵ showed that the 2½-sec seizure delay method on the Four-Ball apparatus correlated better with practice than the S.A.E. machine.

With the introduction of the universal and truck-type hypoid lubricants the position of the S.A.E. machine has undergone a change which nobody appears at the moment to have had the time to explain. The fact that with the universal type a load-carrying capacity of 325-lb scale pull (minimum) is allowed, whereas the older "active" sulphur types were required to carry 550 lb (minimum) is as yet unexplained.

It must not be thought from the above remarks that the development of the machine is at a standstill. The standard test-pieces for the machine are U.S.A. Timken test-cups, T 48651, which are made from nickel-molybdenum steel, S.A.E. 4620, case-hardened to 65-C Rockwell. The test-cups bearing the same number produced in this country are made from a chromium steel. Furthermore, a pool of cups is maintained by British Timken from which test-cups for the Timken machine may be drawn, and used cups sent back for regrinding, the cups being reground until they reach a minimum diameter. If these latter are used on the S.A.E. machine it may be found that some are smaller than the nuts which are to lock them into position. Tests carried out using the different rings

show that a lead soap—"active" sulphur lubricant which carries full load with American cups will only take about 250-lb scale pull using those manufactured in Britain. A universal-type lubricant which fails at 340 lb with American cups will take full load when tested with the British variety. This experience is confirmed by work of the S.A.E. Sub-Committee which has experimented with test-pieces of different steels. Other modifications have been tried in America, such as test-cups of $\frac{1}{4}$ in and $\frac{1}{8}$ in width (instead of the usual $\frac{1}{2}$ in), but the results were unsatisfactory and tended to bunch together.

One of the biggest criticisms of the S.A.E. machine is that there is no temperature control of the oil. Also, since only the bottom cup dips in the oil, and the top one is only cooled by splash or by the oil carried round by the bottom cup, it becomes very hot, especially if the load carried is high: even although the test only takes about 1 minute. (It is worth noting that at full load practically the whole of the output of the 10 h.p. motor is being absorbed in turning the machine.) This fact prompted a criticism of the test as applied to the "active"-type hypoid lubricants. "If the cup is discoloured—*i.e.*, the Brinell is changed—the oil will run to the limit of the machine. Gears are not allowed to change in Brinell, but this appears to be required in the S.A.E. machine. Many lubricants which pass in service are not good enough by the laboratory machine."⁴² That the cups do soften is shown by an observation made by Garton⁴³ that it is possible to roll out an S.A.E. ring to 50 per cent greater diameter without scoring. The writer can endorse this statement, and has even improved on the 50 per cent.

Wear tests at low loads have been investigated in the U.S.A., but even after some hours the loss in weight of the cups was negligible. The S.A.E. sub-committee have also experimented with different surface finishes, including superfinish. With rings finished to the latter criterion it was found that the load carried was increased, and also that the results with different oils bunched together and the test was less discriminating. Sherman⁴⁴ has reported results of tests carried out with two different finishes—*i.e.*, 16–18 and 1–2 microinches. An oil which failed at 117 lb with the coarser, carried 217 lb with the finer finish.

CORRELATION OF RESULTS OF THE VARIOUS TEST MACHINES.

Several writers have given results of investigations showing the readings obtained on various machines and how these correlate with practice. Thus, McKee and co-workers¹⁴ and Neely¹³ give results obtained with the Almen, Timken, Floyd, and large "General Motors" machines; Miller,¹ the Almen, large "General Motors," Timken and S.A.E. machines; van der Minne,²⁴ the Timken, Floyd, Four Ball and S.A.E. machines; Prutton and Willey,⁷ the Almen, Falex, Timken and S.A.E. machines; Blok,³⁵ the Four Ball, Timken and S.A.E. machines; and Prutton and Willey¹⁵ and James,¹⁶ the Almen, Timken, S.A.E., Falex, and Four-Ball machines; the results obtained by Prutton and Willey are given in Tables I and II and those quoted by James in Table III. Van der Minne²⁴ and Van Dijk and Blok²⁹ have given a detailed comparison of the conditions under which the Four-Ball, Timken, Floyd, S.A.E., and the Almen machines

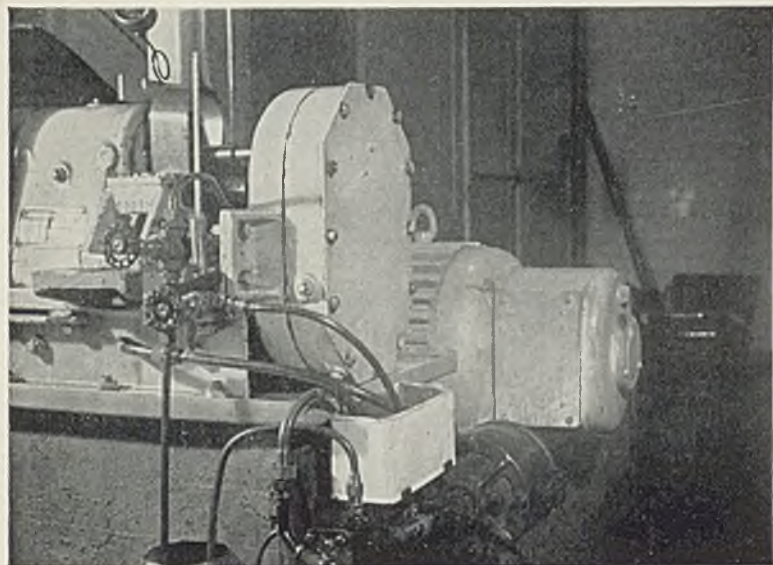


FIG. 2.

TEMPERATURE CONTROL ARRANGEMENT FOR THE S.A.E. MACHINE (FRONT VIEW).

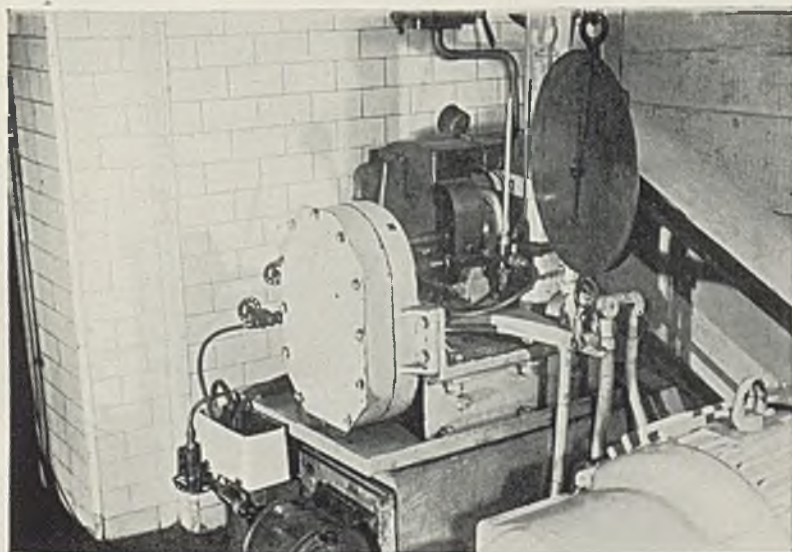


FIG. 3.

TEMPERATURE CONTROL ARRANGEMENT FOR THE S.A.E. MACHINE (BACK VIEW).

[To face p. 214.]

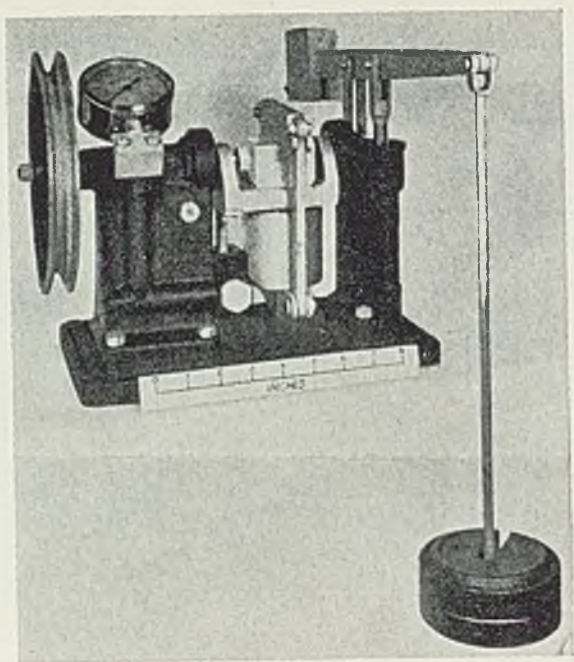


FIG. 4.
OIL-SAMPLE CUP FOR ALMEN MACHINE.

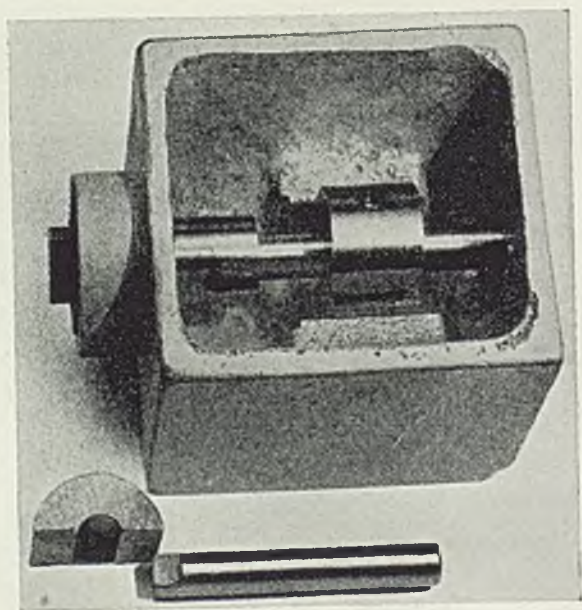


FIG. 5.
ALMEN EXTREME-PRESSURE LUBRICANT TESTING MACHINE.

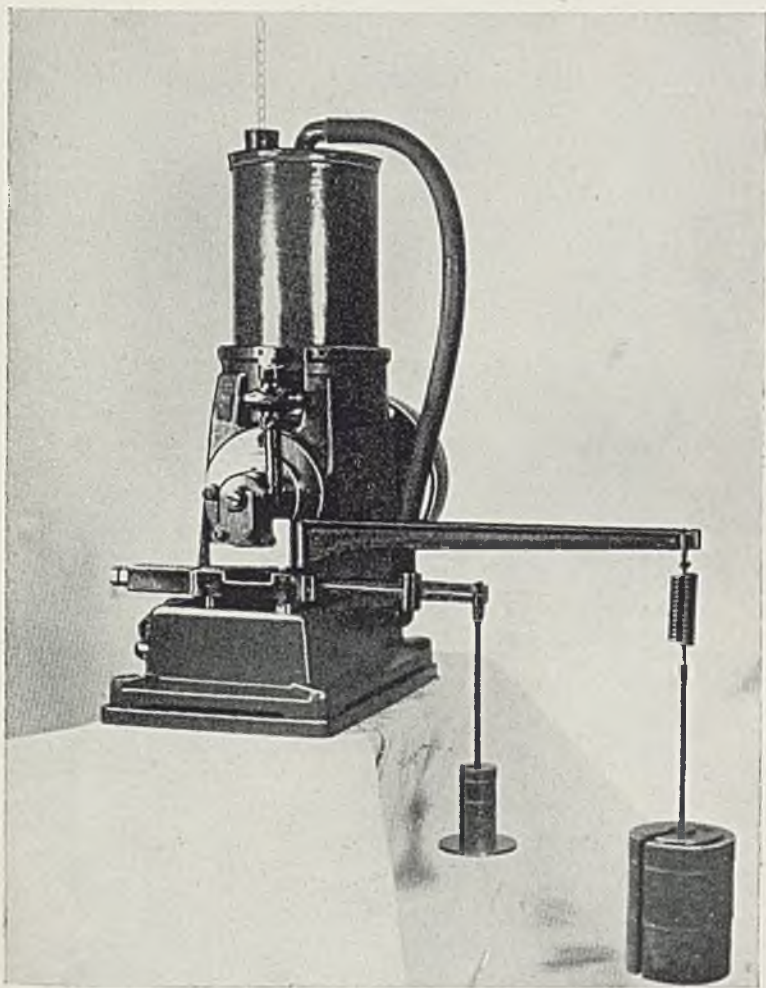


FIG. 6.

TIMKEN WEAR AND LUBRICANT TESTING MACHINE.



FIG. 6a.
THE "FOUR-BALL" PYRAMID.

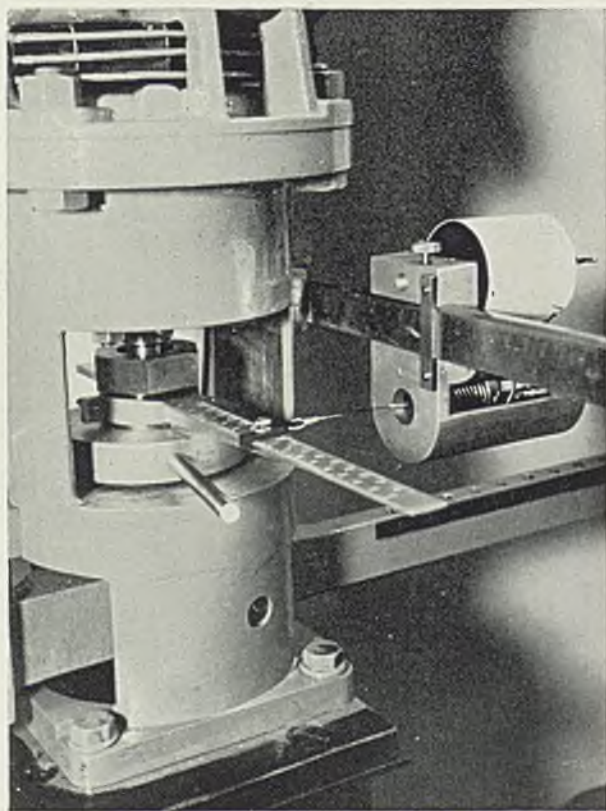


FIG. 7.
CLOSE-UP VIEW OF THE "FOUR-BALL" MACHINE.

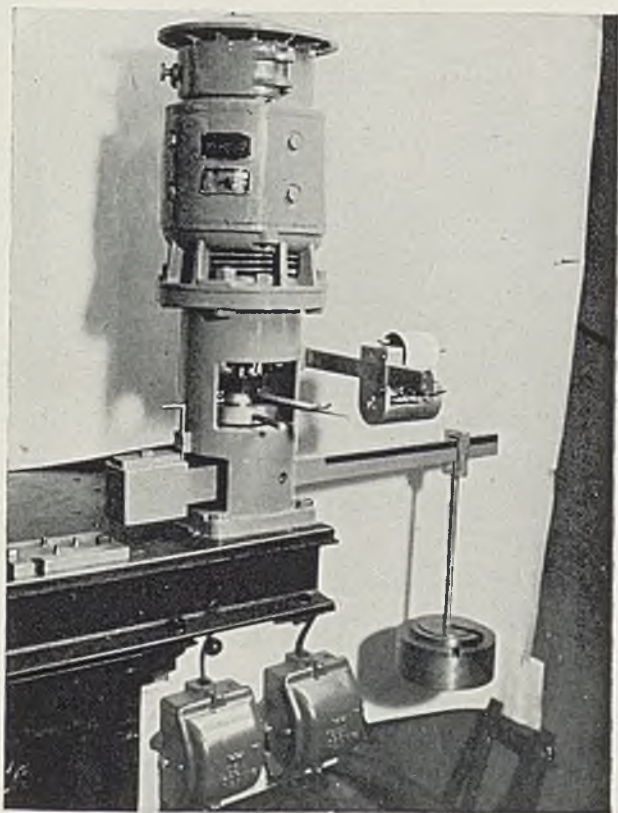


FIG. 8.

THE "FOUR-BALL" MACHINE WITH ELECTRIC MOTOR DRIVE.

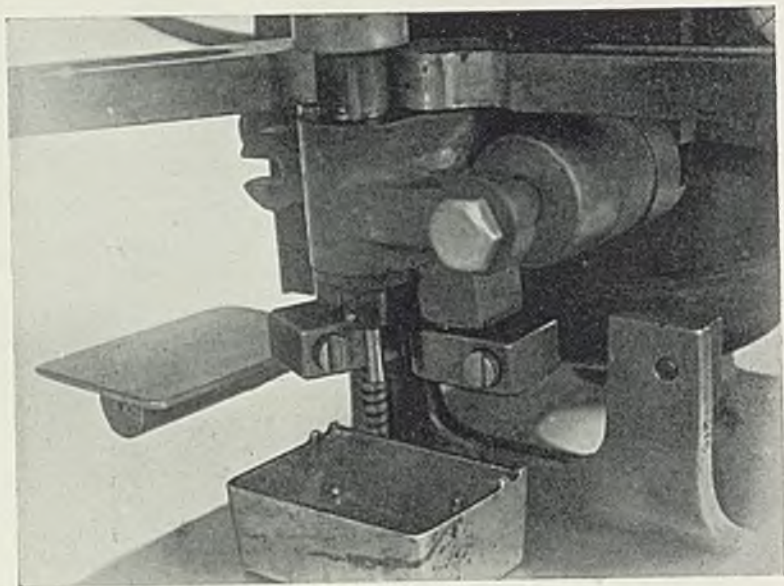


FIG. 9.

THE "FALEX" MACHINE. CLOSE-UP VIEW SHOWING THE TEST PIECES.

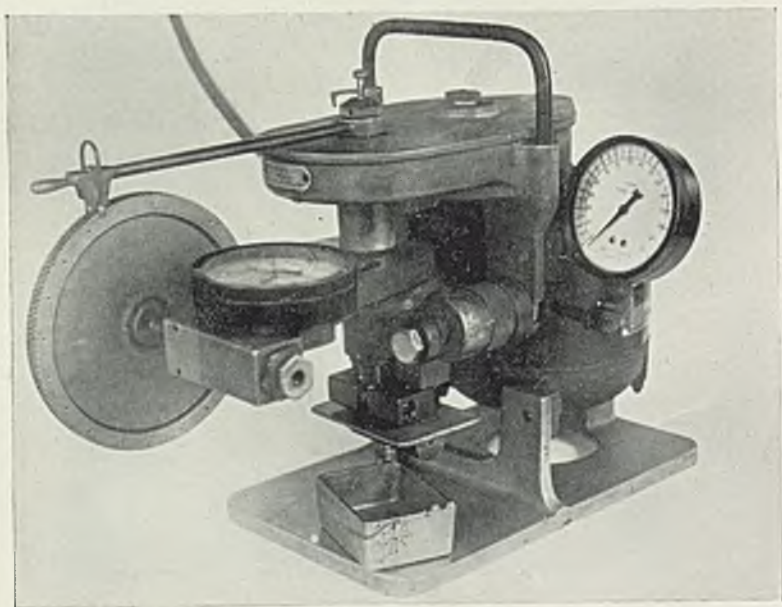


FIG. 10.

THE "FALEX" MACHINE.

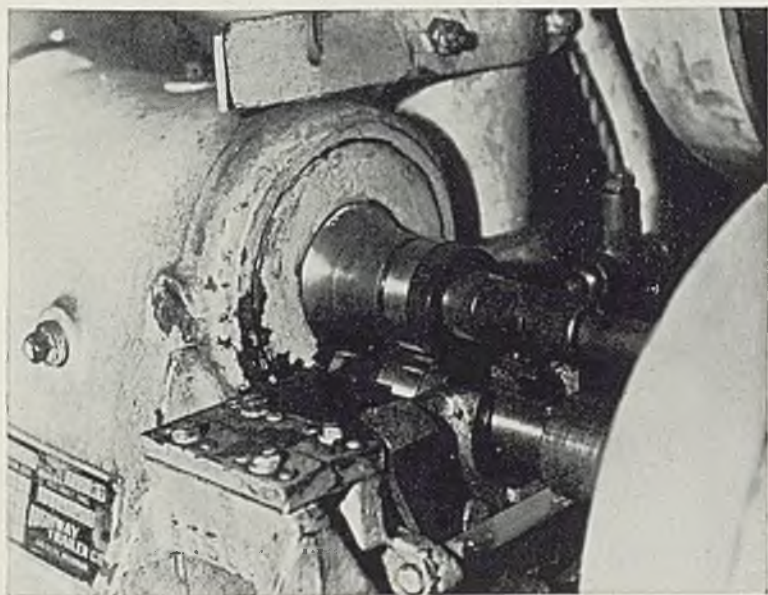


FIG. 11.

CLOSE-UP VIEW OF THE S.A.E. MACHINE SHOWING THE TEST PIECES.

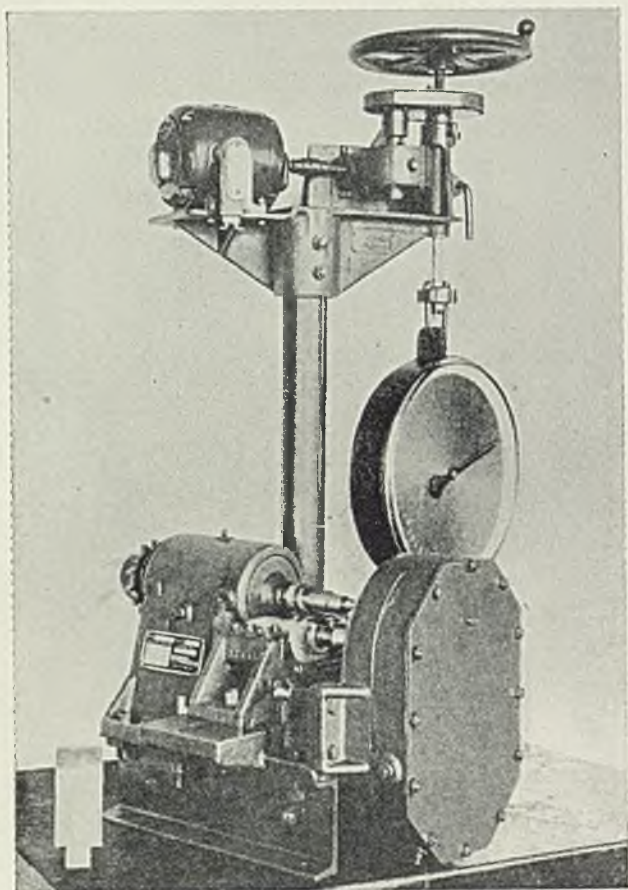


FIG. 12.

S.A.E. EXTREME-PRESSURE-LUBRICANT TESTER.

operate, and contrast these with conditions obtaining in a hypoid gear; a summary of this data is given in Table IV.

When it is considered that no two of these machines operate under the same conditions, it is not surprising to find that only a very rough, if any, correlation exists between them, and this only when each machine is operated under the optimum conditions. It is unfortunate from a practical standpoint that the correlation with service is also not of a high order. It would appear, however, that the S.A.E. machine is giving the best results at the moment.

DISCUSSION.

When the machines are operated under the usual test conditions given in the Appendix, a certain sub-division into types of test is possible. The Almen, Timken, Four-Ball, and Falex machines all incorporate the action of one metal sliding on another. Of these four, the Falex test is a wear test and the Almen a continuous-loading test. Both the Timken and the Four-Ball tests are shock-loading tests. The S.A.E. machine, although a continuous-loading test, differs from the others by virtue of the rolling and sliding action.

Continuous-loading tests on machines with rubbing action only can be criticized on the grounds that wear takes place, reducing the load per unit area; this is particularly marked with certain oils. The shock-loading tests can be criticized on the grounds that no machinery is operated without previously being run-in, and Baxter and his co-workers²² have shown what effect this can have on the load carried, especially when active-type oils, such as E.P. lubricants, are being tested; the remarks above about wear also apply. These results are given in Table V.

Wolf⁴⁵ has stated that in a hypoid gear the unit pressures may be as high as 400,000 p.s.i. whilst sliding velocities may reach 1800 ft/min or higher. The manufacturer's acceptance tests for the universal type of hypoid lubricant consist of a "shock test" and an endurance test at high temperature and load. Bearing these facts in mind, it can be seen that the shock-loading tests as given by the Four-Ball and Timken machines fall short in respect of the sliding velocity, and the latter also in the load. Van Dijk and Blok²⁹ have suggested that the high intensity of pressure in the Four-Ball machine may compensate for the low sliding velocity, and McKee and co-workers,¹⁴ Southcombe, etc.,²³ van der Minne²⁴ and Clayton³⁴ have shown the effect of increasing the rubbing speed. It is possible that with some such modification as indicated the correlation of test results with practice using these two machines could be improved.

None of the machines described has adequate, if any, temperature control of the lubricant. James¹⁶ gives figures for the "levelling off" temperatures at various speeds of different types of American cars, the figures ranging from 120 to 201° F. (the air temperature being 100° F.) and up to as high as 300° F. may be reached in bus and truck operation.¹ It would appear that one of the essential modifications necessary to all the machines is that the tests should be conducted at some temperature similar to that prevailing in service. Since the S.A.E. machine is designed to simulate the conditions of a gear, it might be worth while to see how tests similar to the service tests noted above could be simulated. If a

spray of oil on to the cup is accepted, it should be possible, and has been done in the U.K. to control the oil temperature within reasonable limits. A shock-loading test, after suitable running-in, could then be arranged, as well as a high-load, high-temperature endurance and wear test, the latter to simulate the truck hypoid. In Figs. 2 and 3 is shown the arrangement used for supplying oil at constant temperature to the S.A.E. machine. The oil is taken from the sump and pumped through a heating or cooling coil immersed in a water-bath, and thence to a jet at the back of the test-piece—*i.e.*, as they are coming out of mesh (see Fig. 3). (The oil-cup is fitted with a collecting device so that it is run on a "dry sump" basis, excess oil splashing over the cup being collected from the well of the machine and returned to the sump.) The rate of flow is controlled by the pressure set up by the oil passing through a calibrated

TABLE I.
Summary of Gear Test Ratings.⁷

Lubricant type.	Continuous load.			Shock load.		
	Series I.	Series II.	Series III.	Laboratory.	Road.	Gear test rating.
1. Lead soap Active sulphur	Borderline	Borderline	Borderline	No score	No score	Pass
2. Lead soap Inactive sulphur Chlorine compound	Score	Borderline	Score	Score	Score	Fall
3. Inactive sulphur Saponifiable oil	Borderline Excessive wear Excessive temp.	Score Excessive temp.	Erratic Excessive temp.	Score	Score	Fall
4. Sulphur chloride Saponifiable oil	No score	No score	No score	No score	Erratic	—
5. Chlorine compound Sulphur compound	No score	No score	Borderline	No score	No score	Pass
6. Mineral oil	Score	Score	Score	Score	Score	Fall

orifice, the pressure regulation being effected by a by-pass returning oil to the sump. The temperature of the ingoing and outgoing oil is measured, and gives an indication of the efficiency of the lubrication and the tendency towards seizure. In view of the potent effect of the metal used on the results obtained, all test-pieces should be fabricated from the same alloy as that used in manufacturing the gears, and also in the same condition of heat treatment, hardening, etc. This would require a good deal of co-operation from the gear manufacturers, but since they would benefit by the saving in axles used for tests, it should be worth while.

If a suitable machine or machines could be developed, they would have applications to other fields of gear lubrication besides the hypoid gear. One great difficulty is in finding out how a gear is operating—*i.e.*, the relation of rolling to sliding, the loading and velocities. Merritt⁹ gives a method of calculating tooth pressures and surface velocities, stating, "It is necessary to present in simple form a method of reducing gear tooth loading conditions to first principles in order that the design of

laboratory apparatus for further investigation can be definitely related to actual conditions instead of giving, as happens with some lubricant testing machines, misleading or conflicting results." This latter remark

TABLE II.

*Results of Film Strength Machine and Performance Tests.**

Lubricant type.	Almen, lb.	Falex, lb.	Timken, lb.	S.A.E. machine.			Laboratory gear test ratings.
				500 r.p.m., lb.	750 r.p.m., lb.	1000 r.p.m., lb.	
1. Lead soap	} 30 +	4500 +	25	580 +	580 +	580 +	Pass
Active sulphur							
2. Lead soap *	} 16 ±	2500 ±	68 ±	407 ±	133 ±	98 ±	Fail
Inactive sulphur							
Chlorine compound	} 30 +	2375	86	147	96	81	Fail
Inactive sulphur							
Saponifiable oil	} 30 +	4500 +	33	401	92	90	—
Sulphur chloride							
Saponifiable oil	} 30 +	4500 +	59	580 +	480	330	Pass
Chlorine compound							
Sulphur compound							

* Erratic on all film strength machines.

is to some extent justified by the fact that the Falex, Timken, and Four-Ball machines all indicate that the lead-soap "active" sulphur-type E.P. lubricant gives a high rate of wear, whereas Miller,¹ Zwahl,⁴¹ and Garner,⁴⁶ have stated that this is not so. These statements might have to be modified somewhat since the introduction of hypoid axles into trucks; pertinent data are given in Table VI, which refers to tests also mentioned in Tables I and II.

TABLE III.¹⁶

Typical E.P. Lubricant Testing-machine Results.

Lubricant.	Almen, Weights on arm.	Timken, lb on arm.	S.A.E., lb on arm, 750 r.p.m.	Falex, lb load on gauge.	4-Ball, kg load to seize in 2½ sec.
Lead soap	} 30 +	25	580 +	4500 +	190
Active sulphur					
Lead soap	} 16 ±	68 ±	138 ±	2500 ±	130
Inactive sulphur					
Chlorine compound	} 30 ±	59	480	4500 +	145
Chlorine compound					
Sulphur compound	} 6	7	0	500	75
Untreated oil					

Full-scale tests may be vitiated by such factors as the rigidity of the mounting of the axle and gears, the distortion and deflection of the teeth, and other unaccountable differences. This serves to stress more forcibly the desirability of designing a suitable laboratory test where such factors can be more easily controlled.

TABLE IV.

Details of Testing Machines.

Machine.	Moving parts.	Material.	Kind of movement.	Sliding velocity, ft./min.	Specific pressure on contact surface p.s.i.
Almen	Split bushes around revolving shaft	Bushings and shaft made from S.A.E. 2315 cold drawn steel	Sliding	2 × 39.2	15,000
Timken	Block on steel ring	Block and cup, nickel molybdenum steel S.A.E. 4020 carburized and hardened 65° Rockwell C.	Sliding	400	Initial about 85,000 (load 110 lb), final 7100-17,100
Four ball	Ball on three balls	"S.K.F." balls, chromium steel	Sliding	115	Initial about 797,000 (load 600 kg), final 14,200-142,000
Falex	V blocks around revolving shaft	Shaft of S.A.E. 3135 steel heat treated to Rockwell B84-80 and V blocks of S.A.E. 2320 steel	Sliding	10 × 4	4500 (initial)
S.A.E.	Two steel rollers, one on top of the other	See Timken tester	Sliding and rolling	475 (1000 r.p.m.)	284,000
Hypold	—	Hardened alloy steel	Sliding and rolling	Up to 980-1870	Up to about 355,000

TABLE V.²²*Timken Machine—10-minute Tests on Cups Previously Used at a Load of 100 lb in Doped Oil.*

Oil.	Load applied.	State of cup.	Maximum load withstood without breakdown, lb.	Appearance of scar.
Undoped	Standard method	Unused	11	Smooth
		Used	18	"
		Used and washed in water	19	"
Undoped	Before starting	Unused	6	Smooth
		Used	21	"
		Used and washed in water	20	"
Doped	Standard method	Unused	100	Rough
		Used	100	"
		Used and washed in water	100	"
Doped	Before starting	Unused	20	Smooth
		Used	100	Rough
		Used and washed in water	100	"

TABLE VI.⁷
Comparison of Wear Test Data.

Lubricant type.	Almen wear, total mg.	Timken wear, total mg.	Per cent. iron in lubricant after double axle tests.			
			I.	II.	III.	Average.
1. Lead soap	} 38.50	4.0	0.031	0.038	0.054	0.041
Active sulphur						
2. Lead soap	} 1.55	2.8±	0.035	0.014	0.053	0.034
Inactive sulphur						
Chlorine compound						
3. Inactive sulphur	} 1.50	1.6	0.280	0.610	0.029	0.306
Saponifiable oil						
4. Sulphur chloride	} 4.00	2.2	0.042	0.034	0.031	0.036
Saponifiable oil						
5. Chlorine compound	} 1.50	2.0	0.016	0.021	0.029	0.022
Sulphur compound						

CONCLUSION.

Throughout this paper the lubrication of rear-axle drives has been stressed, and the relation of the testing machines to this particular usage emphasized; also various modifications to these machines to bring them more into line with practice have been indicated. It should not be assumed that this in any way detracts from the use of the various machines in fields of lubrication other than that of gears. With the exception of the S.A.E. machine, it is probable that the machines each serve a very useful purpose in other branches of lubrication technique. Also it is possible, in the future, that it will be found that tests on more than one machine will give the correlation desired.

It is believed that this is the first time that the information given in this paper has been published collectively. Extraction of results from the various papers mentioned has been limited, as anyone interested would be better served by consulting the originals than in reading any extracts which might be made from them. Furthermore, such extracts would make the present review unwieldy and cumbersome. Any mention of, or discussion on, the mechanism by which the various E.P. additives enable hypoid gears to function has been purposely avoided, since this subject would require a paper to itself, and that a very lengthy one.

It is significant that most of the work on this subject has been carried out in the U.S.A., but, since additives are being manufactured in the U.K. it would appear desirable that a more lively interest in the subject should be shown here.

It is hoped that the information contained in this *résumé* of existing thought on hypoid lubrication will prove of value to members of the Institute and others interested, and, if it promotes discussion and progress in this field, it will have served its purpose.

ACKNOWLEDGMENTS.

It is desired to acknowledge the assistance given by other members of the Mechanical Tests Panel in supplying descriptions of the testing technique for use with some of the testing machines mentioned.

APPENDIX.

THE ALMEN MACHINE.

Description.

This machine employs a $\frac{1}{2}$ -in-diameter drill-rod journal and a $\frac{1}{2}$ -in split bushing made from S.A.E. 2315 cold-drawn steel. Both the journal and bushing are used in the unheat-treated "as received" condition. The journal is polished, and the bushing, after splitting, is ground on the bearing surface with a form grinding-wheel. A 0.007-in clearance is provided between the journal and the normal diameter of the bushing, the two halves of the bushing being prevented from rotating by locating pins. In Fig. 4 the test-pieces can be clearly seen, together with the drive and the manner in which they are located in the oil-sample cup, which, with the cradle and hydraulic-loading cylinder holding, respectively, the top and bottom halves of the split bush, is pivoted around the centre of rotation of the journal. The torque developed is indicated through a hydraulic system on a pressure-gauge. The load is applied by adding weights to a pan attached to a lever system; the load on this system being transmitted hydraulically to the hydraulic loading cylinder holding the bottom half of the split bush. The machine is belt-driven at a journal speed of 600 r.p.m., the journal being coupled to the main shaft by a flexible (Oldham) type coupling. The general arrangement of the complete machine, with an indication of its size, can be seen from the photograph in Fig. 5.

Method of Test.

New test-pieces are inserted in the machine and the oil-sample cup filled with the oil to be tested. The motor (or other drive to give 600 r.p.m.) is started, and then 2 lb weights are applied to the loading beam at 10-sec intervals. The test is continued until seizure occurs or until a total of 30 lb has been added (the limit of the machine). Each 2-lb weight is equivalent to 1000 p.s.i. projected bearing area.

Specifications Requiring this Test.

General Motors Corp. Specn. No.	Almen test, Minimum load required.
4588M	12 lb
4520M	30 lb
	Diluted with 70 per cent. mineral oil
	16 lb
4636M	16 lb
4645M	16 lb
4637M	16 lb

THE TIMKEN MACHINE.

Description.

The test-pieces used in this machine are a test-cup or ring (designated T48651), which is tapered on the inside and is parallel on the outside (this is virtually the outer race of a Timken roller-bearing, except that the outer surface has a special finish) and a test block $\frac{1}{2}$ in square by $\frac{3}{4}$ in long. Both test-pieces are made of steel, case-hardened to 60° C. Rockwell, and

ground. Newly ground faces on both pieces are necessary to ensure uniform results, and in no instance must the finish of the test components be altered.

A heavy mandrel is rigidly mounted in Timken tapered roller bearings so arranged as to hold it rigidly in alignment; at one end is mounted a pulley, and it is tapered at the end opposite the pulley to receive the tapered test-cup. The latter is held firmly in place by a circular nut which screws on to the mandrel. The test-block, which is mounted below the cup, is carried by a system of two levers, one above the other, the upper being known as the load lever and the lower as the friction lever. The upper, which carries the test block-holder, is pivoted on a knife-edge mounted in the lower lever. The latter is also pivoted on a universal knife-edge and provided with a stop at the unloaded end. The friction lever is provided with a vernier scale and a sliding weight for obtaining accurate measurements, and both levers are supplied with weight-pans. By virtue of this arrangement, the test-block is always in the same relative position to the revolving cup, and the unit loading over the length of the two test-pieces is always constant. The test-block is inserted in a notch in the holder and held firmly in place by a wedge. Fig. 6 gives a picture of the complete machine, showing the lever systems and the method of applying the weights.

Immediately above the housing carrying the mandrel is mounted the oil-container, holding about $\frac{1}{2}$ -gallon of lubricant. Provision is made for heating the oil and holding it at temperatures up to 250° F. In operation, oil from the container flows over the test-piece to a sump in the base. It is then pumped back by means of a small pump located in the base, and driven from the testing mandrel. For testing greases, the oil splash-guard surrounding the test-pieces is filled with grease before commencing the test. The guard is then kept filled by continually adding grease by means of a grease-gun. To make grease tests comparative, care has to be taken to feed the grease at a constant rate.

The machine requires an electric motor or other source of power of 2 h.p., and the speed of the mandrel is usually 800 r.p.m., giving a rubbing speed of 400 ft/min. (N.B.—The material used for the test-cups produced in the U.S.A. was a nickel-molybdenum steel, whereas those produced in the U.K. were of a chromium steel. This may be subject to alteration. There is probably a slight difference in surface finish.)

Method of Test.

(a) *Load-carrying Ability.* The oil reservoir is filled to within 3 inches of the top, and then heated to the required temperature, usually 100° F. While preparing the test-pieces, the oil is circulated and the motor started to warm up the pump and the mandrel. The test-pieces are prepared by lightly polishing with 1F rouge paper, and are weighed to the nearest milligram, and the necessary weights put on the spring loading-pan ready to place on the machine. The machine is assembled, the cup placed on the mandrel, and the test-block in the holder, and all knife-edges adjusted till in perfect alignment. The test-cup and block are smeared with the lubricant under test.

The oil is started circulating and the machine started and brought up

to 800 r.p.m. The load is applied instantaneously, and not gradually built up. The machine is allowed to run 10 minutes unless a score is noticeable; if no evidence of breakdown is found, the friction load is taken. The motor is then stopped, the flow of lubricant turned off, and oil temperatures taken (a) in the tank and (b) on the block. On dismantling, if there is no sign of pick-up or breakdown of the oil film, the test-block is turned round to a new face, a new cup taken as before, and the procedure is repeated. Initially, the load is increased in 10-lb increments, but once the seizure is bracketed, 1-lb increments are used to find the exact seizure load. The scar width is measured using a Brinell microscope, and the used test-pieces reweighed; the coefficient of friction can also be calculated.

When testing greases, the special screw-down grease gun is fitted in place of the oil-container over the heating elements, and the same procedure is followed as when testing fluid lubricants, care being taken that a constant feed of grease is maintained over the whole 10-minute period of the test.

(b) *Abrasion and Wear Test.* This test is only applied to extreme-pressure oils and greases, the machine being run with a 33-lb lever load for periods of 2 to 6 hours. The test-cup and block are weighed before and after the test, and the combined loss in milligrams is taken as a measure of the abrasiveness of the lubricant. (The combined loss is taken because with some lubricants containing free inorganic matter, such as graphite, asbestos, etc., an excessive wear of the cup is produced.) The grease or oil used for the abrasion test may be re-tested for its load carrying capacity.

Specifications Requiring this Test.

Timken E.P. grease is required to carry a load of 43 lb (minimum) on the load-lever arm in the load-capacity test, and an abrasion test at 33 lb load for 2 hours is run. The load-capacity test is repeated on the used sample from the abrasion test, and should show little reduction in load carrying ability.

THE FOUR-BALL MACHINE.

Description.

The test-pieces used in this machine consist of standard $\frac{1}{2}$ -in steel balls, S.K.F. (Skefko) of ball-bearing quality. One of these is clamped in a chuck at the bottom of a heavy vertical driving-hub, which is mounted on two journal and one thrust-ball bearings. This ball is spun in the cavity formed when three other $\frac{1}{2}$ -in balls are squeezed tightly together by means of a clamping ring, and maintained in position in the bowl filled with the lubricant to be tested, the clamping ring being pressed down by a clamping nut; the position of the balls during the tests is that given in Fig. 6a. The bowl is free to rotate, being mounted on a thrust ball-bearing. A vertical load is applied to the top ball by means of the pressure exerted by the three underlying balls, which are themselves forced upwards by means of weights hung on a counterbalanced lever. During rotation this load causes a frictional torque on the bowl, which can be measured by means of a spring and recording drum actuated by a calibrated arm attached to the bowl. The maximum weight of 40 kgm

can be hung from the farthest notch of the loading lever, and is then equivalent to a vertical load of 800 kgm.

The hub is rotated at a speed of 1500 r.p.m., either by means of a $1\frac{1}{2}$ -h.p. A.C. or D.C. motor, or provision can be made for a belt drive. In the latter case belt-slip must be avoided.

A close-up view of the machine showing the torque measuring arrangements is given in Fig. 7, whilst the complete machine, showing the method of applying the load, can be seen in Fig. 8.

Method of Test.

For each test a set of four new balls should be used; these should be cleaned before being inserted. If grease is to be tested, it is advisable first to fill the bowl with the grease into which the balls are then embedded. The grease is then smoothed over a few millimetres above the top of the three balls. The predetermined load is placed on the lever arm, the motor switched on and allowed to run for 1 minute, and a friction-time chart is obtained for each load applied. Immediate seizure is apparent from the violent displacement of the torque indicator of the machine at the start of the run. Delayed seizure is indicated by a momentary increase in friction at some period during the 1-minute run; if no seizure occurs there will be no increase in friction. The distinction between no seizure and delayed seizure is more accurately determined by examination of the wear-spot after the run.

A series of 1-minute runs are made at varying loads using four new balls and a fresh sample of grease for each test. The wear-spot on the lower three balls is measured, two measurements of each spot being made, one parallel to the axis of rotation of the shaft and the other at 90° from the first. The arithmetical average of the six measurements is taken as the wear-spot diameter. A wear-load diagram is prepared, plotting the wear-spot diameter in millimetres as ordinate versus the axial load in kilograms for each run, on log-log paper. A straight line is drawn through the points from runs in which no seizure occurred, and a second line, almost parallel, through points of immediate seizure; a third line, intersecting both the others, is drawn through points of delayed seizure. At least three points on each line of the curve shall be determined. The lower intersection, between no seizure and delayed seizure, has the co-ordinates D_1 (diam in mm) and L_1 (load in kg); the upper intersection, between delayed seizure and immediate seizures, the co-ordinates D_2 and L_2 . From these points the "Pressure Wear Index" is calculated from the formula:—

$$\text{P.W.I.} = \frac{L_2 - L_1}{(D_2)^2 - (D_1)^2}$$

Specifications in which this Test is Required.

The U.S. Army-Navy Aeronautical Specification No. AN-G-10 requires that the Pressure Wear Index shall be 15.0 minimum. (N.B.—Although the description given here of a test for grease is the only one which has been issued in a specification, the machine is usually used for oil testing and rarely for greases.)

FALEX MACHINE.

(Previously known as the Cornell or Faville-Lovally machine.)

Description.

The test-pieces used in this machine consist of a $\frac{1}{4}$ -in diameter journal of S.A.E. 3135 steel heat-treated to a Rockwell hardness of B 84-86 and two V blocks of S.A.E. 2320 steel. The steel journal is held in a vertical shaft by means of a brass shearing-pin inserted through a hole in the rod, the vertical spindle being gear-driven at a speed of 290 r.p.m. by an electric motor of approximately $\frac{1}{4}$ -h.p. capacity. Two lever loading arms, separately pivoted behind the vertical spindle, have self-aligning recesses in them to take the V blocks, the V notches being vertical and clamped round the steel journal. A close-up view of the machine, showing the test-pieces in their respective positions, is given in Fig. 9. The load is applied by a spring-loaded mechanism consisting of a horizontal spindle with right- and left-hand threads at opposite ends. At the centre of this spindle is a large ratchet wheel which, on rotation, increases the load by squeezing the two lever loading-arms together. The load applied is registered on a loading dial which records the actual load applied to the V blocks. An eccentric driving a pawl can be used for turning the ratchet wheel, and so applying the load at a standard rate; it may also be turned by hand if desired. The torque set up by the rod rotating against the V blocks is indicated by a hydraulically operated pressure-gauge, graduated in p.s.i., the V-shaped bearing blocks, and the entire load-applying mechanism being free to turn about the axis of the test journal.

The two V blocks and the journal are immersed in oil in a metal oil-cup, which is supported on a metal tray incorporating an electric heater. In Fig. 10, which shows the complete machine, the oil-cup has been removed from its normal position to show the position of the test-pieces when a test is being carried out.

Method of Test.

New test-pieces are used for each test. The machine is first warmed up by running at 500-lb load with any E.P. oil capable of withstanding this load for a period of 15 minutes. After warming, the machine is washed off with solvent, new test-pieces are installed, and the cup filled with the oil to be tested. The motor is started and the machine run for 3 minutes, a load of 500 lb being applied mechanically. At the end of the 3-minute period the load is increased mechanically to 1000 lb and maintained for 1 minute, noting any abrupt changes in the torque reaction, since a sudden increase indicates film rupture, and may be accompanied by scoring of the test surfaces or shearing of the test-piece or brass pin. The load is then increased mechanically to 1500 lb, the pawl disengaged, and the ratchet wheel marked. The run is then continued for 10 minutes. Any wear which lowers the load below 1475 lb is taken up by rotating the ratchet wheel until the load is restored to 1500 lb. The number of teeth wear on the ratchet wheel and the time elapsed to the failure point, if any, are noted, together with details of the torque indicator and the maximum temperature developed in the oil-bath. The bushings and test-pin are

examined for type of failure, type of corrosion, and width of scar, all of which indicate some characteristics of the oil under test.

An oil capable of withstanding a load of 1500 lb for a period of 10 minutes with a total wear not exceeding fifty teeth may be considered a good E.P. oil, providing that the stability is sufficient for service requirements and corrosion not excessive.

Specifications Requiring this Test.

The Ford Company's specifications Nos. M 568 A, Summer; B, Winter; C, Tractor summer; D, Tractor winter.

THE S.A.E. MACHINE.

Description.

The test-pieces used are two Timken test-cups T48651 (see Timken machine for details). These are mounted one above the other, both rotating, but in opposite directions, the load being applied by pressing the bottom cup against the top.

The horizontal mainshaft is rigidly mounted on two Timken tapered roller bearings, and terminates at one end with a taper, on to which one of the test-cups is locked by means of a circular nut; the other end carries the driving-pulley. This mainshaft is coupled by means of an Oldham coupling to another shaft protruding from a gearbox. By means of this gearbox, and using different change-wheels, it is possible to drive the secondary shaft, which carries the second test-cup, at reduced speeds of 3.4 : 1, 10.4 : 1, 14.6 : 1, and 20.7 : 1 in relation to the mainshaft. The secondary shaft, which runs in two half-bearings in the oil-box, is coupled to the gear-box by means of a second Oldham coupling. The oil-box, which is free to swing on knife-edges in the direction of the axis of the shafts, is carried in a cradle, which is hinged to a support by means of thin, spring steel. The test-pieces and their location in relation to the oil-box are shown in a close-up view of the machine in Fig. 11. The loading lever, giving a 10 : 1 advantage, is pivoted on knife-edges, and exerts a vertical force upwards, also through knife-edges, on the cradle carrying the oil-box. By this means the test-cup on the secondary shaft is pressed against that of the mainshaft, the Oldham coupling allowing the two shafts to assume a parallel position. By means of a screw adjustment it is possible to shift the support of the cradle, carrying the oil-box and secondary shaft, to and fro in order to align the two test cups.

The end of the loading-lever is connected to a vertical lead screw through a spring-balance, the load being applied by winding up the lead-screw. Provision is made for applying the load mechanically by a separate motor raising the lead-screw, the speed being suitably reduced by a gear-box. The spring-balance is calibrated in pounds, therefore the load actually applied is ten times the scale pull. Fig. 12, which gives a view of the complete machine, illustrates clearly the general layout and the method of applying the load; the drive to the machine has, however, been omitted. More details of the machine can also be seen in Figs. 2 and 3.

The mainshaft is driven at various speeds, but the most usual are 1000, 750, and 500 r.p.m. If an electric motor is used for the drive it should be

of at least 10 h.p., the speed being adjusted by suitable pulleys and, for preference, with a multi V belt drive. Any other motive power of suitable horse power can be used.

Method of Test.

Two new test-cups are used, one being screwed on to the mainshaft, the other on to the secondary shaft. The machine is then completely assembled, and the alignment of the two cups is checked and adjusted, if necessary. The secondary shaft is then taken down and oil poured into the oil-box until it just overflows into the half-bearings. The secondary shaft is then placed in these half-bearings and rotated to ensure that the bearings are adequately lubricated. The machine is then completely assembled and the splash-guard and fume-extractor placed in position. This latter accessory is necessary, as with oils taking high loads the fumes are so dense as to prevent observation of scoring.

The lead-screw is adjusted for mechanical loading at the lowest scale load possible. The driving-motor is started and, at the end of a 30-sec running-in period, the loading motor switched on, the load being applied continuously at the prescribed rate. The lubricant is judged to have failed when scoring of the top test-cup is observed. The scale reading at this point is noted, and the two motors are immediately switched off to avoid damaging the machine. The result is expressed as the scale-pull in lb at which scoring was observed, the conditions under which the test was made being stated.

It is sometimes difficult to observe the commencement of scoring, and as an aid to this it is convenient to have a well-focused light shining on the top ring. The reflection from the ring is quite bright, and makes the observation easy.

If no scoring takes place, the loading motor is switched off at a load of 580 lb (the maximum is 600 lb), to avoid overloading the spring balance. Such results are recorded as "scale-pull 580 plus". A switch can be included in the machine which will automatically switch off the loading motor at 580-lb scale pull.

Tests are sometimes made on the oil samples as received and again after a specified oxidation test.

Specifications Requiring this Test.

All tests are made using the 14-6 : 1 rubbing ratio and 83.5 lb/sec loading rate.

General Motors Spec. No.	Sample.	Mainshaft speed (r.p.m.).	Minimum scale pull (lb.).
4654-M)	As received	1000	550
4655-M)	After stability test	500	550
4734-M)	As received	750	350
4735-M)	After stability test	500	300
4661-M)	As received	1000	550
4662-M)	After stability test	500	400
4663-M)	As received	1000	325
4664-M)	After stability test	500	400

Test.	Test limits.		
	80.	90.	80-90
Load-carrying capacity, lb/min :			
Before heating: 1000 r.p.m.	300	325	325
After heating: 500 r.p.m.	400	400	400

THE CHEVROLET "SHOCK" OR "BUMP" TEST.

All hypoid-gear lubricants shall be capable of satisfactorily lubricating a current model Chevrolet hypoid rear axle, without scoring or scuffing, when tested in accord with the following test procedure :—

Section 1.

- (a) Thoroughly clean axle housing and axle shafts, and instal new third member;
- (b) Fill housing to the proper level with the lubricant to be tested;
- (c) Drive 3·8 miles at 60 m.p.h.;
- (d) Drive 3·8 miles at 70 m.p.h.;
- (e) Check axle for noise on a level concrete pavement, on the drive and coast, at 10 to 40 m.p.h. and 40 to 10 m.p.h. in high gear;
- (f) Drive 38 miles at 75 m.p.h.;
- (g) Check noise as in item (e);
- (h) Drain lubricant into a clean container. Remove axle housing cover and inspect the gears.

Section 2.

- (i) If the gears have not scored under the high-speed driving conditions outlined in Section 1, replace the axle-housing cover and refill with the lubricant, which was drained in accord with item (h);
- (j) Drive car in high gear to 70 m.p.h. Disengage clutch and turn off ignition. Permit car to coast to 65 m.p.h., and immediately engage clutch as rapidly as possible with transmission in high gear and with ignition turned off. With clutch still engaged and ignition off, permit car to decelerate to 40 m.p.h.;
- (k) Repeat item (j) six additional times;
- (l) Check noise, as in item (e);
- (m) Repeat item (j) eight additional times, making a total of fifteen decelerations in high gear;
- (n) Check noise, as in item (e);
- (o) Drive car in second gear to 50 m.p.h. Disengage clutch and turn off ignition. Permit car to coast to 45 m.p.h., and immediately engage clutch as rapidly as possible, with transmission in second gear and with ignition turned off. With clutch still engaged and ignition off, permit car to decelerate to 20 m.p.h.;
- (p) Repeat item (o) four additional times;
- (q) Check noise, as in item (e);
- (r) Repeat item (o) five additional times, making a total of ten decelerations in second gear;

- (s) Check noise as in item (e);
- (t) Remove third member and inspect gears;
- (u) If the gears show no indication of scoring or scuffing on inspection (item (t)), a second third member is installed and the axle refilled with fresh lubricant, and the entire test is repeated.

Examination of the gears may be made at any time during the test procedure if the axle becomes objectionably noisy, or if scoring or scuffing is suspected.

DYNAMOMETER OR ENDURANCE TEST.

Mount in a rear-axle dynamometer a complete axle-housing from a Chevrolet $1\frac{1}{2}$ -ton truck, fitted with 6 : 37 ratio hypoid gears. Check the gear back lash, which must be within 0.006-0.014 in. Fill the axle-housing to the proper level with the test lubricant. Run in for 30 minutes under friction load only, gradually raising the speed to 324 r.p.m. on the propeller shaft, and then follow the procedure outlined below. Cooling of the lubricant in the differential is accomplished by a water-spray applied to the outside of the carrier and rear axle housing.

- (a) 175° F. maximum lubricant temperature, and 324 r.p.m. Operate for successive 30-minute periods with the following torques on the propeller shaft : 168, 378, 588, 800, and 1000 lb-ft;
- (b) With lubricant temperature of 185° F., speed of 324 r.p.m., and 1157 lb-ft torque on the propeller shaft, operate for 300,000 pinion revolutions, unless scoring or other failure occurs. Inspect the ring gear-tooth surfaces at the completion of each 75,000 pinion revolutions;
- (c) With lubricant temperature of 210° F., speed 324 r.p.m., and 1157 lb-ft torque, operate for 25,000 pinion revolutions and inspect the ring gear-teeth;
- (d) Under the above load and speed, but with a lubricant temperature of 235° F., operate for 25,000 pinion revolutions and inspect the ring gear-teeth;
- (e) Finally, with the same load and speed, but with a lubricant temperature of 260° F., operate for 25,000 pinion revolutions and inspect the ring gear-teeth.

The lubricant is considered to have passed if no failure occurs during the test and if the gear teeth are not scored at the completion of the run at 260° F. lubricant temperature.

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METALS AND ALLOYS IN THE PETROLEUM INDUSTRY.*

By JOHN H. WILSON.

INTRODUCTION.

IN spite of the fact that man has employed metals for his purposes since a very remote period in history, it is only in comparatively recent years that he has investigated their internal structure, or has tried to improve their qualities by alloying and by thermal and mechanical treatment. It is not intended to retrace the obscure, but nevertheless interesting, history of the development of metals, but only to describe briefly the properties of some of the metallic materials available to the petroleum industry at the present time.

The term metallic materials is used as the term metals, in the title of this paper, is perhaps a little misleading. The term metals should be used only when referring to pure metals and pure metals are seldom used commercially, though there is an increasing tendency towards the use of greater purity in industry generally.

The petroleum industry is almost entirely dependent on metallic materials for the construction of equipment for winning its raw material and for processing the many subsequent derivatives. The unique properties possessed by metals of high melting point combined with great mechanical strength and, when correctly chosen, of resistance to corrosion, render such materials very suitable for making plant for the storage or preparation of the various and numerous products obtainable from crude petroleum.

The petroleum industry cannot be classed as a large consumer of metals in the same sense as, say, the shipbuilding or motor industries. It is, however, a large user of metals, and vast quantities of metallic materials are in use in various forms in the oilfields and refineries throughout the world.

The progress made by metallurgy continues to elucidate, where possible, the problems arising from the use of metals and to devise new alloys and metal treatments based on the fundamental properties of the metals themselves. Unfortunately, long periods of time sometimes elapse between successful laboratory experiments and the stage where new materials are available to industry. Such delays may be caused by scarcity of raw material or by difficulty in extracting the required metal from its ore. Platinum and tungsten are examples of metals whose industrial availability was delayed for many years because their melting points were beyond the limit of the existing melting facilities. It is interesting to note that both these metals have, at some period in their history, been prepared by the method known as "powder metallurgy."

To-day a large range of alloys is available to constructors, and the number continues to grow. Some of the modern alloys are of complex composition and demand close attention to their subsequent thermal and mechanical

* Paper read before Stanlow Branch, Institute of Petroleum, on Nov. 8, 1944.

treatment in order that their special properties may be fully developed. It should be clearly understood that the heat treatment of certain alloys—the medium and high alloy steels, for example—is a very precise operation demanding a thorough knowledge of the principles involved from those who carry it out.

From time to time one hears accounts of alloy parts which have failed in service prematurely, or which have proved impossible to work satisfactorily. In a great many such cases the underlying cause has proved to be unsuitable thermal treatment resulting from lack of appreciation of the proper requirements.

It is as well to bear in mind the fact that with the introduction of more highly developed alloys greater demands will be made on the ability of those who fabricate them and, no less, on those who use them.

FERROUS ALLOYS.

Mild Steel.

Far and away pre-eminent to all other metals is iron, familiar to everyone in the form of either wrought iron or steel, which is an alloy of iron with a small amount of carbon. The term "steel" is used to cover a whole range of alloys from "dead" mild steel, with a carbon content less than 0.25 per cent, to highly alloyed varieties containing large amounts of chromium, nickel and, perhaps, other elements.

Little need be said of common mild steel. Its frequent appearance in such familiar wrought shapes as girders, wire, tubes and sheets is sufficient testimony of its ductility, mechanical strength and general ease of working. Mild steel also possesses good castability and may be readily moulded into large and intricate shapes by regular foundry practice.

It is only proposed to mention two types the properties of which depend on the way in which they have been cast into ingots prior to being wrought into sheets, bars or tubes.

The impurities, soluble and insoluble, in a melt of steel tend to segregate in the interior portions of the ingots into which the metal is cast. Selective freezing, aided by the ebullition caused during the escape of gas (produced by the reaction of FeO and carbon) in the solidifying metal, is responsible for this peculiar segregation phenomenon.

Ingots of this type of steel have an impure core with an envelope of purer metal on the outside. Such steel is known as "rimming" steel on account of the ridge which forms on the head of the ingot during cooling. A small addition of metallic aluminium, or other suitable metal, is sometimes made to the steel just prior to pouring. The effect of the aluminium is to de-oxidize the metal, which then solidifies more evenly, with less segregation than in the case of "rimming" steel. This is known as "killed" steel.

Sheet or bar rolled from ingots with impure cores will have impurity in the central parts, although the outer skin may be relatively pure. It sometimes happens that highly stressed parts are erroneously made from "rimming" steel, with serious results. For instance, lap-welded tubes are frequently made from "rimming" steel and do in fact show greater resistance to corrosion owing to the greater purity of their interior and exterior walls. The failure of this kind of tubing frequently occurs when

deep threading is carried down into the impure core. It is desirable to avoid the use of "rimming" steel for stressed parts, and to use the "killed" variety for such purposes.

The corrosion resistance of plain carbon steel is not of a very high order. Nevertheless, when correctly specified and properly treated, its life is such as to render it the most widely used metallic material under many mildly corrosive conditions.

Typical Properties of Mild Steel.

Tensile strength	25 to 29 tons p.s.i.
Yield strength	11½ to 13 tons p.s.i.
Elongation on 8 in.	25 to 30 per cent.

High Tensile Steels.

The tensile strength of plain carbon steels increases with the carbon content, but not without some sacrifice of ductility and impact strength. Owing to the consequent lowering of other desirable mechanical properties, the addition of carbon is not the usual method employed for increasing the tensile strength of plain carbon steels. The addition of small quantities, about 0.7 to 1.0 per cent, of such elements as manganese, silicon, nickel and chromium to low-carbon steels has the effect of increasing their tensile strength and ductility. These low-alloy steels are known as "high-tensile steels" and are employed where high yield strength is required from cheap steel. The corrosion resistance of "high-tensile steels" is little better than that of mild steel although, under certain conditions, it may be superior, owing to the fact that they are usually made from purer materials. The ordinary grades of "high-tensile steel" are of low carbon content. They are therefore not hardenable by thermal treatment. They are used in the annealed or normalized condition.

Typical "Low-Alloy" Alloy Steels.

No.	C, per cent.	Mn, per cent.	P, per cent.	Si, per cent.	Cu, per cent.	Cr, per cent.	Ni, per cent.	Elastic limit, p.s.i.	Tensile strength, p.s.i.	Elongation on 2 in., per cent.	Reduction in area, per cent.
A	0.08	0.27	0.145	0.8	0.41	1.01	0	60,000	78,000	40	72
B	0.08	0.43	0.145	0.16	1.07	—	0.54	56,000	71,000	38	60
C	0.22	0.60	0.045	0.25	0.92	—	1.98	54,000	91,000	30	54
D	0.37	1.14	0.022	0.84	—	0.50	—	80,000	125,000	23	58.
36	0.21	0.43	0.008	—	—	—	—	38,000	57,000	42	64
SS	0.2	0.5	0.03	0.15	—	—	—	25,000/ 30,000	50,000/ 60,000	25/30	—
CS	0.45	—	—	—	—	—	—	45,000	85,000	12	—

High-tensile steels are used where it is desirable to reduce the weight of structures. They are being employed in increasing amounts in the fabrication of welded equipment, while their application to highly stressed parts is well known.

Medium Alloy Steel.

Next in the list of alloy steels stand the "medium alloy steels," the alloy content of which may vary from 2 to 6 per cent. There are many steels falling into this class, but it is proposed to deal with only two. The addition

of 3 per cent chromium to 0.25 per cent carbon steel produces an alloy with high tensile strength, high ductility and resistance to "creep" at moderately high temperatures. The 3 per cent chromium steel is widely used in the petroleum industry, especially where gases at high pressures have to be handled. Although this steel is not markedly corrosion resistant, generally speaking it is valuable, on account of its resistance to the embrittling effect of hydrogen at elevated temperatures and pressures.

The 4 to 6 per cent Cr—0.5 per cent Mo steel is very well established in the petroleum industry, where its high creep strength and resistance to oxidation at elevated temperatures render it particularly suitable for making still tubes.

The 4 to 6 per cent alloy is not resistant to the attack of sulphur compounds and wastage may occur where turbulence either removes entirely, or at least damages, what sulphide films are able to form on the surface of the metal. It is not uncommon for 4 to 6 per cent still tubes to have a life six times as long as plain carbon steel tubes in similar service. It should be remembered that both the 3 per cent and the 4 to 6 per cent chrome steels have pronounced air-hardening properties and call for rigid adherence to the specified heat-treatment procedure.

The heat and corrosion resisting steels depend largely on chromium content for their special qualities. The addition of large amounts of chromium to iron results in the production of a rather brittle alloy, and attempts to remedy this defect have led to the development of alloys containing several other alloying elements.

Stainless Steel.

It is unfortunate that the term "stainless steel" should be so frequently used to designate, indiscriminately, a large number of highly alloyed steels with widely varying degrees of corrosion resistance. In Great Britain the term "stainless steel" is often applied to the 12 to 14 per cent chrome alloys, while the name "Staybrite" is generally reserved for the 18-8 austenitic variety. The standard work on stainless steel published in America (*Stainless Steels* by Thum), deals with a wide range of alloys with chromium contents from 3 to 35 per cent. S. L. Hoyt, another American authority, in his recent data book, deplores the use of the term "stainless steel" and even questions the propriety of applying it to the 18-8 alloys. The confusion of the trade names of such alloys frequently results in the misapplication of one or other grade, and failures sometimes occur. The selection of resistant alloys is a scientific matter calling for great care and experience backed by actual plant tests. The only satisfactory scheme to adopt when specifying or ordering these alloys is that based on the actual chemical composition of the material under consideration.

Of the many grades of so-called stainless steel the well-known 18 per cent Cr-8 per cent Ni is too familiar to need lengthy description. The straight 18-8 alloy, although very satisfactory for many purposes, fails to meet the requirements of certain conditions, and in order to overcome the difficulties titanium, tungsten and molybdenum are added in amounts of some 1 to 4 per cent. Titanium is used to stabilize any small amount of carbon which may be present and likely to result in intergranular corrosion,

while tungsten improves the mechanical properties of the alloy at elevated temperatures. Molybdenum confers a considerable degree of resistance to sulphuric acid on 18-8 alloy.

"Modified Analysis" Stainless Steels.

Element.	Per cent added.	Result.
Al	4.0 to 4.5	Greatly improves resistance to scaling.
Cu	1.0 to 4.0	Increases resistance to HCl.
Cb	0.6 to 1.5	Increases resistance to granular attack.
Mn	3.0 to 5.0	Stabilizes. Improves ductility.
Mo	1.0 to 4.0	Increases resistance to H_2SO_3 and H_2SO_4 .
Se	0.2 to 0.3	Imparts free machining properties.
Si	0.5 to 3.5	Increases resistance to high temperature scaling.
S	0.3 to 0.4	Improves machining properties.
Ti	0.4 to 0.8	Strong stabilizing action.
W	2.5 to 3.5	Stabilizes strength at high temperature.

General.

Specific uses for these alloys are not given because of the different results obtained from identical alloys under similar conditions.

Carbon steels offer a wide range of mechanical properties at room temperature but they lose their valuable characteristics at elevated temperatures, with the result that the mechanical properties tend to seek equilibrium and the effects of previous heat treatment are lost. Wherever plain carbon steels prove unsatisfactory at elevated temperatures, alloy steels should be used. Such heat-resisting steels may contain small additions of chromium or silicon, but frequently 12 to 27 per cent chromium is necessary with or without further large amounts of silicon or nickel.

Although nitrogen is known to be detrimental to plain carbon steels, it has been found to be very useful in preventing excessive grain growth in high chromium steels which are exposed to high temperatures for prolonged periods. Thus nitrogen ranks as yet another alloying element for heat-resisting steels.

The more recent developments in the petroleum industry have taxed the ingenuity of metallurgists, and although much work has been done, with a view to improving heat-resisting alloys, much still remains to be accomplished in this field.

A useful and widely used alloy of this class is that known as "Immaculate 5." This is an austenitic alloy with 25 per cent Cr and 20 per cent Ni. "Immaculate 5" is sometimes used in the fabrication of reversing dampers for catalytic cracking furnaces.

In the case of catalytic cracking (dehydrogenation) the surface of the tubes holding the catalyst are subjected to alternate periods of carburization and oxidation, with the result that a pitting type of corrosion takes place in spite of the high chromium alloy used (27 per cent Cr).

A most unfortunate consequence of high chromium addition to iron is the poor mechanical strength of the resulting alloy. Extreme brittleness is often exhibited by these alloys when they are cooled to about 475° C and below. In this condition they are extremely "notch sensitive" and cases

are known where machining lines and pipe wrench-marks have led to extensive cracking.

Casting alloys, with good resistance to the effects of exposure at high temperature, are of particular interest to petroleum refiners who require materials suitable for tube hangers and other furnace accessories. Here again, chromium, nickel and silicon are the chief alloying elements employed for securing the desired resistance to creep and oxidation.

A well-known alloy, often used for tube hangers, is called "Callite," of which three grades with the following compositions are made :—

S.28	.	.	3 per cent Ni	31 to 35 per cent Cr)	}Balance iron.
B.28	.	.	10 to 12 per cent Ni	28 to 32 per cent Cr)	
N	.	.	65 to 68 per cent Ni	16 to 19 per cent Cr)	

Aluminium bearing cast irons, containing up to 7 per cent Al, are useful for high-temperature work, although some are very brittle.

Cast Iron.

The alloys classed as cast irons form a large group of materials with widely varying properties, and anything like a detailed description of even a few individual compositions is outside the scope of this paper.

The chief difference between the cast iron and the carbon steels is the much higher carbon content of the former. Steels have a maximum carbon content of about 1.5 per cent. In cast irons the carbon content is rarely below 2 per cent, and sometimes as high as 4.5 per cent.

The carbon in cast iron may be present as graphite, cementite Fe_3C , austenite, which is a solid solution of carbon in gamma iron, or as a eutectic mixture of cementite and austenite. The constitution and structure of the cast irons are, in fact, very complicated because these depend not only on chemical composition, but to a large extent on the rate of cooling.

The type of cast iron most frequently used for the bedplates of machinery, pump bodies and condenser water-boxes is grey cast iron. Grey cast iron has a coarse granular structure made up of large flakes of graphite set in a ground mass of pearlite. The latter is a peculiar structure produced by the lamella arrangement of layers of cementite and iron. The structure of the common grey cast iron may be varied within limits by adjustments in chemical composition and rate of cooling. Grey cast irons are soft, readily machinable, and fairly resistant to mild corrosive conditions, especially to atmospheric corrosion.

When greater resistance to wear is required a cast iron containing a larger proportion of cementite is prepared by altering the composition, usually by reducing the silicon content and also by rapid cooling. The process known as "chilling" has been known from early times, and is still largely practised. Chilling may be carried out by casting the molten metal into cold iron moulds, or by placing pieces of iron in sand moulds so as to produce a local hardening effect on the resulting castings.

The plain white cast irons are very hard. They are usually cast to shape and then ground to size. Such irons are used for lining chutes handling coal, coke or other abrasives, and often give good service where corrosion and abrasion have to be combated.

In recent years the use of alloy cast irons has steadily gained favour, and

to-day there is a very comprehensive range of useful materials from which to choose. The chief alloying elements used for making special cast irons are silicon, chromium, nickel, copper and molybdenum. Their effects are briefly as follows :—

Si . . .	Promotes the formation of graphite.
Cr . . .	Is a strong carbide former.
Ni . . .	Is a graphite former and increases the stability of the austenite.
Cu . . .	Stabilizes the austenite and increases strength and corrosion resistance.
Mo. . .	Is a mild carbide former, but increases the tensile strength. 0.5 to 1.5 per cent Mo increases the tensile strength by 60 per cent.

Of the alloy cast irons only a few can be briefly dealt with here. The heat- and corrosion-resisting alloy "Ni-Resist" is an austenitic iron—*i.e.*, the carbon is present as a solid solution in gamma iron—and may be compared with the austenitic stainless steels. Gamma is not stable at atmospheric temperature unless large amounts of alloying elements are present and so "Ni-Resist" contains from 12 to 15 per cent Ni, 5 to 7 per cent Cu and 1.5 to 4.0 per cent Cr. "Audcoloy" is very similar in composition and is largely used for making valves and cocks.

Before leaving the cast iron, something should be said of an alloy known as "Meehanite," which is a high-strength cast iron made by a process called inoculation. By taking a melt of iron which would normally solidify as white iron—*i.e.*, all iron carbide—and adding to it silicon in the form of calcium silicide as it is being poured, the formation of graphite is promoted with a consequent increase in strength of the resulting metal. "Meehanite" is a high-duty cast iron. It is not markedly heat resistant or resistant to severe corrosion conditions. Some grades of "Meehanite" contain alloying elements—*e.g.*, silicon—in a considerable amount, but none of these is austenitic. The superior mechanical properties of "Meehanite" make it suitable for many purposes where extra strength is required, and it has proved satisfactory for making trays for fractionating columns. A considerable saving in weight can be effected because such trays can be cast thinner for equivalent strength.

Some Cast Irons.

	C, per cent.	Si, per cent.	P, per cent.	Mn, per cent.	Cr, per cent.	Ni, per cent.	Mo, per cent.	Cu, per cent.	S, per cent.	Tensile strength, tons.
Grey cast iron	3.25	2.2	0.2	0.6	—	—	—	—	0.13	11
Machinery, heavy	3.25	1.2	0.35	0.5	—	—	—	—	0.18	12
Cast iron valve bodies	2.6	2.2	0.08	0.7	—	1.1	—	—	0.08	28
Hard white iron 600 B.H.	2.5	1.0	0.2	0.5	1.5	4.5	—	—	0.1	—
"Ni-Resist"	2.8	1.5	0.1	1.5	3.5	14.0	—	7.0	0.2	—
"Nicrostal"	1.8	6.0	—	1.0	—	18	—	—	—	—
<i>High duty :</i>										
"Meehanite"	2.8	1.4	—	—	—	—	—	—	—	23
"Ni-Tensyl"	2.6	1.5	0.1	—	—	2.0	—	—	—	28
Ford crankshaft	1.5	0.7	0.1	0.4	0.5	—	—	2.0	0.08	49
Internal combustion en- gine cylinder	3.3	2.25	0.21	0.8	0.9	0.2	1.0	1.0	0.13	22

HIGH SILICON IRON ALLOYS.

The interesting silicon iron alloy known as "Tantiron" or "Duriron" is less familiar to those engaged in the petroleum industry. The composition of this material is approximately 14 to 15 per cent Si—0.8 per cent C

and the balance iron. When properly made it resists the attack of strong mineral acids to a remarkable degree, as the mineral acids form a thin layer of silica on its surface which prevents further attack from taking place.

Unfortunately the mechanical properties of "Tantiron" are very poor indeed, and this limits its use to relatively small parts which are not subjected to shock. "Tantiron" is too hard to machine with ordinary tools, and is extremely brittle. In spite of the severe limitations to its use "Tantiron" occasionally proves to be the only solution to the acute problems arising out of the pumping of concentrated acids. In an attempt to avoid the machining difficulties a method for coating mild-steel pump-rods with a thin layer of "Tantiron" was developed at Stanlow and has proved satisfactory, although further development is still required.

Another variety of silicon iron is HCL Metal, and in this alloy the silicon content is about 12 per cent with 6 per cent of copper. This material resists hydrochloric acid to a marked degree, but its mechanical properties are similar to those of "Tantiron."

The alloy "Cronite" is worthy of mention in connection with material suitable for acid pumps. This alloy was originally developed for the manufacture of heat-resisting carburising boxes and other furnace accessories. "Cronite" can be obtained in both cast and wrought forms, is easily machined, and possesses great mechanical strength.

Typical Composition of "Cronite."

63.5 per cent Ni, 13.7 per cent Cr, 20.3 per cent Fe, 1.04 per cent Mn, 0.43 per cent Si, 0.19 per cent S, 0.8 per cent Al.

NON-FERROUS METALS.

The non-ferrous metals are not suitable for the construction of large pieces of equipment, chiefly on account of their higher cost, but also because their mechanical properties are of a lower order than those of steel.

Copper base alloys, such as the brasses and the bronzes, are the most important non-ferrous alloys used in the petroleum industry, where they find wide application in the form of pump parts, and tubes and tube plates for heat-exchange equipment.

The commercial brasses fall into two main groups: (a) those with less than 38 per cent Zn and (b) those with between 38 and 45 per cent Zn. These are known as alpha and alpha beta brasses respectively. Copper alloys containing more than 45 per cent Zn are hard and brittle and have little commercial application.

Soon after the introduction of the steam engine, copper and copper-base alloys were employed for making condenser tubes for steam plant, and even to-day the brasses with slight modifications are the materials chiefly used for this purpose. The high thermal conductivity of copper is the reason for its use in this connection.

Among the brass condenser tube alloys in use to-day the following are the best known:—

- 70 per cent Cu, 30 per cent Zn.
- 60 per cent Cu, 40 per cent Zn.
- 70 per cent Cu, 29 per cent Zn, 1 per cent Sn.
- 76 per cent Cu, 22 per cent Zn, 2 per cent Al.

Muntz
Admiralty brass.
Sometimes called "Alumbro"
or "Yorcalbro."

All these alloys give good service under suitable conditions, although they are liable to undergo corrosion peculiar to the water side, and this matter has been the subject of much research in the past.

It should be remembered that all copper-rich alloys are rapidly attacked by sulphur compounds, and this fact renders condenser tubes liable to failure under some conditions. Increasing Zn content confers some resistance to attack by sulphur compounds on the brasses, but at the same time it increases the susceptibility to waterside corrosion.

Muntz or 60 per cent Cu-40 per cent Zn brass, and 70 per cent Cu-30 per cent Zn, brass condenser tubes are seldom used for refinery purposes owing to their unsatisfactory behaviour in sea water or contaminated water. Both Admiralty brass and "Alumbro" were developed as a result of attempts to overcome waterside corrosion, and the superior properties of these alloys depend on the formation of a film of oxide on the surface when they are immersed in water.

Typical Analysis of Monel Metal.

	Ni, per cent.	Cu, per cent.	Fe, per cent.	Mn, per cent.	Al, per cent.	Si, per cent.
B	67	30	1.4	1.0	—	—
K	66	29	0.9	0.4	2.75	—
S	63	30	2.0	0.5	—	4.0

These Zn-rich alloys undergo a peculiar form of corrosion known as "dezincification" when they are placed in contact with electrolytes such as sea water. High temperature greatly facilitates this attack. It has been found that the addition of a small amount of arsenic or antimony will lessen the liability to this form of attack, and tubes containing about 0.05 per cent As are now on the market. From the results of actual plant experiments it appears that the 76-22-2 (Alumbro) is superior to Admiralty brass, although under some conditions the difference is not appreciable.

Before leaving the copper brass alloys attention should be directed to a group of alloys marketed under various trade names, although in fact they are alpha brasses containing small amounts of other elements such as nickel, silicon, aluminium, and manganese. These alloys are widely advertised as possessing corrosion resistance to a marked degree and such statements may lead one to suppose that they can be used for handling strong acids and other corrosives, which is not the case. It is only fair to state, however, that some of these alloys have superior resistance to atmospheric and marine conditions, while at the same time they often combine greater mechanical strength with better casting and working properties.

Of the large number of alloys which have a more limited application in the petroleum industry those which contain a large proportion of nickel are worthy of attention. For many years the well-known Monel alloys have been used to combat corrosion in various branches of chemical industry, with great success in many cases. Unfortunately nickel is rapidly corroded by sulphur and sulphur compounds, and this limits, to some extent, the use of nickel-rich alloys in petroleum refining.

The Monel alloys possess high mechanical strength and are suitable for many purposes where a strong corrosion resisting material is required. It is interesting to note that Monel metal appears to be suitable for handling the acid sludge produced during the acid refining of lubricating oils. Monel pump rods, buckets and liners are suitable for service where acid or saline water causes the rapid failure of bronze or steel parts.

Though alloys rich in nickel do not find wide application in petroleum refineries the 80 per cent Cu-20 per cent Ni alloy is often used for "Taylor Ring" gaskets for high-pressure steam service and tubes of the 70 per cent Cu-30 per cent Ni alloy have a remarkably long life in turbine condensers.

SAFETY TOOLS.

The tendency for some metals to yield sparks when struck with hard substances is a matter of considerable interest to the petroleum industry, where the fire risk is ever present. Some metals yield sparks very readily when they are scratched in such a manner that small fragments are detached from the mass. A classical example of this is the ages-old tinder box. Of more recent date is the so-called "flint lighter" which, incidentally, is not made of flint, but of a cerium iron alloy sometimes called Misch metal. The reason for the production of sparks is the rapid oxidation of the fragments of metal, detached iron, and especially hard steel, which readily yield sparks when they are struck, and tools made of these metals are often stated to be the cause of serious fires. The spark-producing property of metals is related to their hardness. Very soft iron may not yield detached fragments when it is abraded or, if it does, the particles may be too large to oxidize rapidly enough to give rise to sparks. Hard steels resist abrasion, and therefore only very small fragments are broken off when they are struck. Hardened tools, such as hammers and chisels, are thus seen to spark quite frequently.

Tools made of soft iron or mild steel are unserviceable, and so high-strength non-ferrous alloys, especially those rich in copper or nickel, are frequently used for making non-spark safety tools.

The tin and aluminium bronzes are often used for this purpose on account of their cheapness and reasonable mechanical strength, although they lack the desired degree of hardness.

The copper, nickel, aluminium alloy known as "Kunial" is a popular material for safety tools, and it is worth considering briefly. The properties of this alloy are dependent on a curious hardening effect caused by the precipitation of the Beta nickel aluminium constituent which takes place when the alloy, which has been water quenched from a high temperature, is subsequently tempered. "Kunial" quenched from 900° C has a hardness of 68 V.D.H. and a tensile strength of 22 tons. After tempering for two hours at 600° C the hardness rises to 194 V.D.H. and the tensile strength to 45 tons.

Copper beryllium alloys exhibit a similar precipitate hardening effect and the alloy, consisting of Cu 97.5 per cent-Be 2.5 per cent, finds some application for making safety tools. It is, however, rather brittle.

The precipitation-hardened alloys require considerable skill in manufacture, and unfavourable reports on their behaviour are numerous. One

case is known where a spanner was broken in the post on its way from the makers.

Brittle "safety tools" can be very dangerous, and hand injuries and falls from scaffolding have resulted from their use.

On no account should cast non-ferrous "safety" spanners be used. They are frequently unsound owing to casting defects, and in the cast state neither "Kunial" nor the bronzes possess the desired mechanical strength. The necessity to ensure that only forged or hot pressed "safety" tools are used is stressed.

In conclusion it should be remembered that the surface of soft metal tools may become loaded with particles of silica, which may cause sparking if they come into contact with steel objects.

JOHN H. WILSON

The late J. H. Wilson was a native of Cornwall but his early life was spent in London, where he received his elementary education at various L.C.C. schools, the last of which he left at the age of fourteen. During the next seven years he received a certain amount of private tuition and this was followed by his taking a full-time course in mineralogy and metallurgy at the Chelsea Polytechnic.

For a time he was employed in the Cornish tin industry, and this association no doubt led to his deep interest in the two subjects which he studied at Chelsea.

After a period with Messrs. Derby & Co. (Metallurgists) he joined the Shell Haven laboratory staff of the "Shell" Refining & Marketing Co., Ltd., in 1937. His interest in refinery metallurgical and corrosion problems very quickly became evident, and it was not long before all such problems were being referred to him for elucidation.

When the extensive development of the "Shell" Refining & Marketing Co.'s Stanlow refinery was taking place in 1939, the need for an experienced metallurgist was felt, and Wilson was so obviously the right man for the job that he was transferred there just before the war. His work at Stanlow was of the highest order, and metallurgical problems which were referred to him were handled in a most capable manner. In 1943 he was given charge of the refinery Inspection Department, for which position his detailed study of refinery corrosion problems made him most suited.

In late 1944 he was seconded on loan to the Heysham refinery of Trimpell, Ltd., which opportunity for obtaining further experience with a new set of refinery metallurgical and corrosion problems was eagerly accepted by him. A few months later, however, he was taken seriously ill and after an operation, from which he at first appeared to be making a good recovery, he died suddenly in February, 1945, at the comparatively early age of thirty-six.

Wilson's principal hobby was his work—the study of metallurgy and corrosion—and most of his spare time was spent in enlarging his knowledge of the subject by reading all the more important publications as they appeared and by discussion of his problems with others. His advancement to the important positions he occupied during the last few years was entirely due to his untiring study of his favourite subject, and his career is one which should act as an incentive to every young man taking up his first position in industry. His kindly disposition and his readiness to help others at all times endeared him to a very wide circle of friends and associates by whom his early demise is most deeply felt.

J. W. V.

